

**COMPARATIVE EVALUATION OF FLUORIDE  
RELEASE AND RECHARGE OF PRE-REACTED  
GLASS IONOMER COMPOSITE AND NANO-  
IONOMERIC GLASS IONOMER WITH DAILY  
FLUORIDE EXPOSURE – AN *IN VITRO* STUDY**

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## CERTIFICATE


This is to certify that this dissertation titled “**COMPARATIVE EVALUATION OF FLUORIDE RELEASE AND RECHARGE OF PRE-REACTED GLASS IONOMER COMPOSITE AND NANO-IONOMERIC GLASS IONOMER WITH DAILY FLUORIDE EXPOSURE – AN *IN VITRO* STUDY**” is a bonafide record of work done by **Dr. John Philip** under my guidance during his postgraduate study period between **2010– 2013**.

This dissertation is submitted to **THE TAMILNADU Dr. M.G.R. MEDICAL UNIVERSITY**, in partial fulfillment for the degree of **Master of Dental Surgery in Branch VIII –Pedodontics and Preventive Dentistry**.

It has not been submitted (partially or fully) for the award of any other degree or diploma.

  
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## **ABSTRACT**

### **BACKGROUND AND OBJECTIVE**

Today there are several fluoride containing dental restorative materials available in the market including glass ionomer cements, resin modified glass ionomer cements, polyacid modified resins (compomers), pre-reacted glass ionomer composite (giomer), and composites which may act as fluoride reservoir, to increase fluoride levels in saliva, plaque and dental hard tissues, or may help to prevent or reduce secondary caries. Fluoride containing dental materials show clear differences in the fluoride release and uptake characteristics which may be influenced by several environmental factors.

The present in-vitro study was conducted to compare and evaluate the fluoride release and recharge of pre-reacted glass ionomer composite and nano-ionomeric glass ionomer with daily fluoride exposure.

### **METHODOLOGY**

Thirty-six specimens each of pre-reacted glass ionomer composite and nano-ionomeric glass ionomer were divided into three treatment groups, namely control group (n=12), fluoridated dentrifice (1000 ppm) once daily group (n=12) and fluoridated dentrifice (1000 ppm) once daily + fluoridated mouthrinse (225 ppm) group (n=12). Each specimen was suspended in a pH cycling system consisting of a demineralizing solution (pH 4.4) for 6 hours and remineralizing solution (pH 7) for 18 hours. Fluoride release was

measured every day for 21 days in both the immersion media using a fluoride ion specific electrode.

## **RESULTS AND CONCLUSION**

The fluoride release of both the restorative materials was found to be higher in the demineralizing solution than in the remineralization solution. Amongst both the materials, the nano-ionomeric glass ionomer had a higher fluoride release than the pre-reacted glass ionomer composite. Also the highest fluoride release was observed from samples which were exposed to both the fluoridated dentrifice and the fluoridated mouthrinse. Both the restorative materials showed no sustained pattern of fluoride recharge. The amount of fluoride released from both the materials decreased over a period of time. However, the nano-ionomeric glass ionomer showed better fluoride rechargability than the pre-reacted glass ionomeric composite.

## **KEY WORDS**

Pre-reacted glass ionomeric composite, nano-ionomeric glass ionomer, daily fluoride exposure, fluoride release and fluoride recharge

## **INTRODUCTION**

Dental caries is a microbial disease of the calcified tissues of the teeth, characterized by demineralization of the inorganic portion and destruction of the organic substance of the tooth.<sup>1</sup> It is still considered to be one of the most prevalent chronic diseases affecting the human race.<sup>1</sup>

One of the key factors in preventing dental caries has been recognized as increasing the resistance of teeth to acid by encouraging the development of a remineralization mechanism on the enamel surface.

The progression or reversal of the caries process depends on the balance between pathological and protective factors. Fluoride has been identified as one of the protective factors, which tilts the caries balance towards the positive side. Earlier it was assumed that the beneficial effect of fluoride was mainly pre-eruptive, strengthening the tooth enamel during development, but it has now become clear that the constant presence of low levels of fluoride in the mouth inhibits demineralization and enhances remineralization of dental hard tissues.<sup>2</sup>

Fluoride is well documented as an anticariogenic agent.<sup>2</sup> A variety of mechanisms are involved in the anticariogenic effects of fluoride, including the formation of fluorapatite that has lower solubility than the original carbonated apatite, the enhancement of remineralization, interference of ionic bonding during pellicle and plaque formation, and the inhibition of microbial growth and metabolism. Fluoride released from restorative materials can

inhibit caries through all these mechanisms, although it seems likely that the enhancement of remineralization is the major mechanism by which fluoride released from restorative material is effective.<sup>3</sup>

The use of fluoride to reduce the frequency of caries can be divided into preventive and restorative categories. Modes of prevention include topical fluoride treatment, fluoridated dentrifice, fluoridated water and devices for controlled release of fluoride. Certain types of fluorides may also be incorporated in restorative materials, which may be beneficial because of the observed cariostatic action of fluoride.<sup>3</sup>

There are several fluoride containing dental restorative materials available in the market including glass ionomer cements, resin modified glass ionomers, polyacid modified resins (compomers), pre-reacted glass ionomer composite (giomers) and resin composites.<sup>4</sup> Fluoride containing dental materials show clear differences in the fluoride release and uptake characteristics and may act as fluoride reservoir to increase fluoride level in saliva, plaque and hard dental tissues, and may help to prevent or reduce secondary caries.<sup>5,6</sup> Short and long term fluoride release from restorative materials are related to their matrices, setting mechanism, fluoride content, nature of fluoride incorporated into resin based materials and also depends on several environmental conditions.<sup>7,8</sup>

A recent development has been the introduction of Pre-reacted glass ionomer (PRG) -composites (Giomers), which employ the use of prereacted

glass ionomer technology to form a stable phase of glass ionomer cement in the restoration.<sup>9</sup> The name "giomer" is a hybrid of the words "glass ionomer" and "composite", which pretty well describes what a giomer is claimed to be.<sup>9</sup> Fluoroaluminosilicate glass in these materials reacts with polyalkenoic acid in water prior to inclusion into silica-filled urethane resin. These materials have fluoride recharge, biocompatibility, smooth surface finish, excellent aesthetics and clinical stability, which have made them popular for restoration of root caries, noncarious cervical lesions, class V cavities and deciduous tooth caries.<sup>10-12</sup>

There is a higher amount of total and free fluoride release from pre-reacted glass ionomer composite when compared with that of compomer and composite resin.<sup>13</sup> It has been said that the extent of glass ionomer matrix of the glass filler plays an important role in fluoride releasing and recharging abilities of the resin based materials.<sup>13</sup> Also it has been shown that pre-reacted glass ionomer composite and compomers do not have the initial fluoride "burst" effect associated with the glass ionomer cements.<sup>14</sup>

There is a great variance in fluoride release among different types of glass ionomer cements probably due to the differences in composition, powder/liquid ratio and mixing time.<sup>15</sup> In addition, it has been noted that decreased physical properties are associated with increased fluoride release.<sup>16</sup> Research into the development of fluoride containing materials, to improve the physical properties of these materials and to provide long term fluoride

release lead to the development of the nano-ionomeric glass ionomer. The nano-ionomeric glass ionomer combines nanofiller and nanofiller clusters with fluoroaluminosilicate glass particles. It is based on the acrylic and itaconic acid copolymers necessary for the glass-ionomer reaction with fluoroaluminosilicate (FAS) glass and water.<sup>17</sup> This new material intends to bring adequate mechanical properties, to enhance aesthetics in terms of smoothness, polishability and precision of shade characterization.

The decrease of fluoride release in the long term is thought to restrict the ability of materials to inhibit secondary caries around restorations because the low levels of fluoride released on the long term may not be at levels that are required for a therapeutic effect.<sup>3</sup> However, it has been reported that glass ionomer cements and pre-reacted glass ionomer composite can take up fluoride from the environments as a means of replacing fluoride that has been lost.<sup>10</sup> The source of fluoride for replacement can either originate from daily low concentration sources like fluoride dentrifices and fluoride mouthrinses or it can also be taken up from the oral fluids at very low concentrations.

With this background, the present study was done to compare the fluoride release and recharge of two relatively new materials i.e. Nano-ionomeric glass ionomer (Ketac N100, 3M ESPE) and pre-reacted glass ionomer composite (Beautifil II, Shofu Inc.) with daily fluoride supplementation.

## **AIMS AND OBJECTIVES**

1. To evaluate the amount of fluoride release and recharge of pre-reacted glass ionomer composite.
2. To evaluate the amount of fluoride release and recharge of nano-ionomeric glass ionomer.
3. To compare the fluoride release and recharge of pre-reacted glass ionomer composite to that of nano-ionomeric glass ionomer.
4. To determine the effect of daily topical fluoride application on fluoride release and recharge from pre-reacted glass ionomer composite and nano-ionomeric glass ionomer.

## **REVIEW OF LITERATURE**

**Forsten L (1977)<sup>18</sup>** in one of the earliest studies compared the release of fluoride from a glass ionomer cement with that from a silicate cement. The test specimens used for the study were shaken and stirred in a solution with hydroxyapatite for 7 weeks. The solution was then changed every week. The fluoride that was taken up by the hydroxyapatite was measured. The highest amount of fluoride was released from the test specimens during each of the first 2 weeks than during each of the subsequent 5 weeks. The continuous release of fluoride had not decreased much with time. The results of the study showed slightly more fluoride release from the glass ionomer cement than from the silicate cement.

**El Mallakh BF, Sarkar NK (1990)<sup>7</sup>** compared the fluoride release of two conventional and two metal-reinforced glass ionomer cements in artificial saliva and deionized water. The amount of fluoride ions released into it from four glassionomer cements were compared with the amount of fluoride ions obtained in deionized water. The glass-ionomer cements used were: Ketac-Fil (ESPE), Ketac-Silver (ESPE), Fuji-II (GC), and Miracle Mix (GC). Ten disc samples (2 cm x 0.1 cm) of each cement were made. Each sample was individually suspended in either de-ionized water or artificial saliva (five samples in each medium). The samples were stored for 24 hours at 37°C and then transferred to



fresh media for a total of 60 days. The collected suspension media were buffered with TISAB, and fluoride content was measured using a ion specific electrode. The data obtained demonstrated that: (1) glass-ionomer cements had a higher fluoride release in de-ionized water than in artificial saliva; (2) Ketac-Fil released 20% more fluoride in saliva than did the amount released by Fuji-II, the latter releasing 49% more fluoride than Ketac-Fil in de-ionized water; and (3) conventional glass ionomers showed a higher fluoride release than did metal-reinforced glass ionomers in both media. The authors however recommended that levels of fluoride release observed in artificial saliva were probably more representative of the materials' actual clinical behavior, since this medium more closely reflects the chemical conditions of the oral environment.

**Forss H, Jokinen, Spets-Happonen S, Seppa L, Luoma H (1991)<sup>19</sup>** conducted to compare the levels of fluoride and mutans streptococci in plaque grown on glass ionomer (Ketac-Fil) and composite (Silar) restorations in vivo. From tunnels left under the brackets bonded either with glass ionomer or composite, plaque samples were collected at 14, 28, and 42 days after bonding. For glass ionomer the mean counts of mutans streptococci in plaque were found to be  $0.5 \times 10^3$ ,  $6.7 \times 10^3$ , and  $8.8 \times 10^3$  colony forming units (CFU) at the first, second, and third collection, respectively. For the composite restorations, the corresponding values were  $32.1 \times 10^3$ ,  $14.6 \times 10^3$ , and  $120.6 \times 10^3$  CFU. For glass ionomer the mean concentrations of fluoride were 19,985, 5,788, and 5,019

ppm during the first, second, and third collections of 14-day old plaque samples, respectively, whereas for composite restorations the mean concentrations of fluoride were about 200 ppm throughout the study. The results of the study indicate that the fluoride level in plaque growing on glass ionomer is much higher than when compared to the fluoride levels in plaque growing on composite restorations. The presence of fluoride seemed to have affected the level of mutans streptococci in present in dental plaque.

**Forsten L (1991)**<sup>20</sup> studied the long-term fluoride release from four glass ionomer cements and one resin composite. The aim was to study the fluoride release 1) from 7 and 15-month-old glass ionomer specimens after treating them with fluoride; 2) from fresh compared with matured material; and 3) from specimens stored for 29 months in running water. Glass ionomer test specimens which had been in running water for first 7 then 15 months were treated with a 50 ppm fluoride solution after which the specimens were again exposed to running water for first 24 hours and then 1 week. The fluoride release was measured after each of the two periods of time. The fluoride treated specimens released more fluoride than the nontreated ones. This effect was not observed with composite resin specimens which were studied for comparison. Fluoride release from fresh glass ionomer specimens was observed to be 3-10 fold compared to specimens that had matured for 3 days. The release of fluoride from specimens that had been in running water for 29 months was measured and the

results were compared with those of earlier measurements. It was found that the release reached a constant level for all tested glass ionomers during the second year.

**Hatibović-Kofman S, Koch G (1991)**<sup>21</sup> determined Fluoride release from glass ionomer cement in vivo and in vitro. The aims of this study were to investigate in vivo the release of fluoride from three glass ionomer cements (GICs) Vitrebond, Ketac-Fil and ChemFil II into the saliva of preschool children for a 1-year period and also to study in vitro the release-absorption-release of fluoride from the same GICs for 16 weeks. In the first part of the study, glass ionomer restorations were placed in primary teeth in preschool children. Unstimulated saliva was collected and the fluoride in the saliva was measured before placement of the restorations, immediately after, after 3 weeks, after 6 weeks, and after 1 year. In the second part of the study, test specimens of GICs were placed in deionized water and the release of fluoride was measured weekly for 16 weeks. At week 12, samples were exposed to fluoride toothpaste. The concentration of fluoride in saliva was 0.04 ppm before placement of the restorations. After three weeks it had increased to 0.8 ppm and the level remained as high as 0.3 ppm even after 1 year. In the laboratory study the tested glass ionomer cements showed a capacity to absorb fluoride from the fluoride toothpaste and then release it. It is concluded that glass ionomer cement can act as a rechargeable slow release fluoride device.

**Takahashi K, Emilson CG, Birkhed D (1993)<sup>22</sup>** conducted a study to determine release of fluoride from 1) discs made from five glass ionomer cements and two composites, and 2) the same discs after exposure to different sodium fluoride (NaF) solutions, were studied. The specimens were placed in distilled water for 10 weeks. After 24 hours and then once a week, the specimens were transferred to fresh distilled water. After 5 weeks, the specimens were divided into four groups and exposed to 0, 0.02, 0.2 and 2% NaF solutions for 5 minutes. The fluoride release was highest during the first week after preparation, after which it decreased sharply and then more slowly. The amount of fluoride released was ordered: liner/base > restorative glass ionomer > composites. The composites released significantly less fluoride than the glass ionomer cements. After exposure to NaF, the fluoride release was significantly higher for the silver cermet material than for the other glass ionomers tested. From a clinical point of view, the results from this study imply that glass ionomer restorations may act as intraoral devices for the controlled slow release of fluoride at sites at risk for recurrent caries.

**Carvalho AS, Cury JA (1999)<sup>23</sup>** conducted a study to determine the level of fluoride released from different restorative materials in different storage solutions. Most of the data reported on release of fluoride from dental materials are based upon measurements made in deionized water and artificial saliva, which do not simulate the dynamics of caries development. So, the purpose of this study was to determine the level of fluoride released from different restorative materials

in storage solutions, considering the caries process (pH-cycling). Six cylindrical samples of each material (Chelon-Fil, Dyract, Variglass, Vitremer, and Tetric) were prepared and suspended individually in 2.0 mL of each studied solution. The studied media were deionized water, artificial saliva, and solutions for pH-cycling (demineralizing solution--pH 4.3 and remineralizing solution--pH 7.0). All solutions were changed daily over 15 days. Fluoride release was determined after buffering the solutions with an equal volume of TISAB. The fluoride release was higher in pH-cycling than in the other solutions ( $P < 0.05$ ), and changes of the rank order of fluoride release from the studied materials occurred when the different media were considered ( $P < 0.05$ ). The data suggest that the comparison of fluoride released from dental materials is dependent on the medium used in the evaluation.

**Karantakis P., Helvatjoglou-Antoniades M., Theodoridou-Pahini S., Papadogiannis Y (2000)<sup>15</sup>** evaluated the amounts and the pattern of fluoride release from one metal-reinforced glass ionomer cement, two resin-modified glass ionomer cements, one compomer, and one composite resin placed in double-distilled water, artificial saliva, and lactic acid. Measurements of fluoride ion release were made for a total of 105 cylindrical specimens (10 mm in diameter and 1.5 mm in height). They were taken over a period of 16 weeks at the intervals of 4, 8, 12, and 24 hours, as well as 2, 3, 7, 14, 28, 56, and 112 days. The pattern of fluoride release was similar for all of the examined materials. The greatest

amount of fluoride was released from the metal-reinforced glass ionomer Argion. The resin-modified glass ionomers Vitremer, Fuji II LC; the compomer Dyract; and the composite resin Tetric followed in ranking order. The pH of the environment strongly affected the fluoride release from the materials. There was a significant difference ( $P < 0.001$ ) in the amounts of fluoride released in lactic acid v/s water and artificial saliva, whereas, there was no significant difference ( $P > 0.05$ ) in the amounts of fluoride released in water versus artificial saliva.

**Dejan LJ, Markovic DL, Petrovic B.B and O Peric T.O (2002)<sup>24</sup>** evaluated the fluoride concentrations at the surfaces of glass-ionomer materials with respect to different storage media and different pH environments. Five glass-ionomer materials, Fuji Triage (FT), Fuji II LC (FII), Fuji VIII (FVIII), Fuji IX GP (FIX), and Ketac N100 (KN), were analyzed in this study. Resin-based fluoride releasing material Heliocore F (HSF) was used as a comparison material. The sample consisted of 120 cured cement disks ( $n = 20$  disks of each tested material,  $10 \times 1.5$  mm). Five disks of each material were stored in 4 different storage media (I- saline, II- acidic solution pH = 2.5, III- acid solution pH = 5.5, IV- NaF solution ( $c = 500/106$ )). After 7 days, two disks of each material were transferred from media I, II and III to the NaF solution for 3 minutes. EDS analysis was conducted in 3 randomly selected spots of each experimental disk. Differences between the experimental groups have been analyzed using Student's t-test with the level of significance set at  $P < 0.001$ . FT showed the highest

fluoride content at the surface of the material. The lowest amounts of fluoride ions were detected at the surfaces of the FT disks stored at low pH environments, and this difference was statistically significant ( $p < 0.001$ ). Glass-ionomers showed significantly higher fluoride concentrations when compared to the HSF ( $p < 0.001$ ). After immersion in the NaF solution, fluoride concentrations at the surfaces of the disks increased when compared with previous storage media (FT>FVIII>KN>FII>FIX). SEM analysis of the surface morphology revealed numerous voids, cracks and microporosities in all experimental groups, except for KN and HSF. The authors observed and concluded that, glass-ionomer releases more fluoride when the environment was at lower pH, leaving less fluoride content at the surface of the material stored at the acidic solution, thus providing the highest amount of fluoride when it is most needed to prevent secondary caries. Fluoride levels increased to five times more after treatment of glass-ionomer specimens with NaF solutions. The major increase in fluoride content was observed in the disks previously stored in acidic solution.

**Itota T, Nakabo S, Iwai Y, Konishi N, Nagamine M, Torii Y (2002)<sup>25</sup>** in a study determined if fluoride-releasing materials can be expected to inhibit the secondary caries. The aim of this study was to evaluate the effect of fluoride-releasing adhesives on inhibition of secondary caries in outer and wall lesions. Two commercial fluoride-releasing adhesives, Reactmer bond (RB) and One-up bond F (OB), and a commercial adhesive without fluoride release,

Mac-bond II (MB), were used prior to placement of restorative materials without fluoride release, Lite-fil II A (LF) and Estelite (EL), and a fluoride-releasing restorative material, Reactmer paste (RP). Class V cavities prepared on extracted human premolars were restored with various combinations of the materials: MB/EL, OB/EL, RB/LF and RB/RP. The restored teeth were incubated in bacterial medium containing sucrose with *Streptococcus mutans* for 14 days. Microradiographs of specimens showed no wall lesions in all groups and an acid-resistant layer adjacent to the restoration in the caries-like lesion. OB/EL, RB/LF and RB/RP groups showed thicker layers than the MB/EL group. The RB/RP group formed the shallowest outer lesion among all groups. These results indicate that fluoride-releasing adhesives are effective in the prevention of wall lesions but exhibit little outer lesion inhibition. Therefore, combined restoration using a fluoride-releasing adhesive and fluoride-releasing restorative material should be selected to inhibit secondary caries.

**Senawongse P, Nilasri K, Okuda M, Otsuki M, Tagami J (2002)<sup>26</sup>** conducted a study to evaluate the rechargeable effect of current fluoride-releasing materials on recurrent caries and caries inhibition zone formation. A fluoride containing composite resin (Reactmer, Shofu), a polyacid-modified composite material (Dyract AP, Dentsply), a composite resin (Clearfil AP-X, Kuraray) and a conventional glass-ionomer material (Fuji IX, GC), were used for the study. One hundred and twenty standardized class V cavities with enamel and dentin margins



on extracted human third molars were prepared, divided into four groups and restored with one of selected materials following the manufacturer's instruction and kept in tap water. The specimens were further divided into 3 groups with different conditions; demineralization for 4 days in an acid buffer solution (pH 4.5) after 7 days storage, after 90 days storage or after recharge at 90 days with 2.2% NaF gel. The specimens were then cut and polished. The depth of lesion ( $\mu\text{m}$ ) and the thickness of inhibition zone ( $\mu\text{m}$ ) were observed under a confocal laser-scanning microscope. The authors concluded that fluoride recharging with NaF gel significantly reduces the depth of the lesions and the effect was more marked with giomers.

**Yap A.U.J, Tham S.Y, Zhu L.Y, Lee H.K (2002)<sup>14</sup>** evaluated the amount and pattern of fluoride release by a giomer (Reactmer), a compomer (Dyract AP), a conventional glass ionomer cement (Fuji II Cap) and a resin modified glass ionomer cement (Fuji II LC). Specimen discs ( $6 \pm 0.2$  mm diameter and  $1 \pm 0.2$  mm thick) were prepared for each material using custom molds. Each disc was placed in 1 ml of deionized water for 24 hours at  $37^{\circ}\text{C}$ . After one day the water was extracted and analyzed. The specimen discs were then re-immersed into another 1 ml of fresh deionized water. This was repeated for 28 days. Fluoride release was determined from sample solutions taken during the first seven days and at days 14, 21 and 28. An initial fluoride “burst” effect

was observed with glass ionomers. The giomer did not show an initial fluoride “burst” effect. The glass ionomers released significantly more fluoride than the compomer and giomer at day one.

**Itota T, Carrick TE, Yoshiyama M, McCabe JF (2004)<sup>13</sup>** evaluated the fluoride release and recharge in giomer, compomer and resin composite. The aim of the study was to examine the fluoride recharging and releasing abilities of resin based materials containing fluoridated glass filler to determine whether the extent of the glass – ionomer matrix of the material affects these properties. Three materials having a different proportion of the hydrogel matrix surrounding the glass filler, namely: Reactmer paste, Dyract AP and Xeno CF, were used for this study. Five disk specimens of each material were placed into distilled/deionized water and the fluoride release measured during 38 days. For fluoride recharge the disks were exposed to 250 ppm fluoride solution for 1 hour and the pre- and post-recharge fluoride release were determined using an ion-selective electrode (total fluoride ions) and ion chromatography (free fluoride ions). The amounts of total and free fluoride release from each material at the initial period in descending order were Reactmer paste > Dyract AP > Xeno CF ( $P < 0.05$  ANOVA and Scheffe's test). After fluoride recharge, Reactmer paste showed a greater amount of fluoride release than the other materials. Dyract AP and Xeno CF showed a similar total level of fluoride release after recharging. For Xeno CF the amount of total fluoride released after recharging was significantly greater than that of free

fluoride, but there was no significant difference between total and free fluoride released after recharging for Dyract AP ( $p = 0.05$  paired t-test). The giomer used in this study thus showed the highest amount of total and free fluoride release during the initial phase of the study and also showed the highest fluoride release after recharge. These results suggested that the extent of the glass-ionomer matrix of the glass filler played an important role for fluoride-releasing and recharging abilities of the resin-based materials.

**Okuyama K, Murata Y, Pereira PN, Miguez PA, Komatsu H, Sano H (2006)**<sup>27</sup> conducted a study to measure the amounts of fluoride release and uptake from fluoride-containing materials before and after daily topical fluoride applications. A conventional glass-ionomer: Fuji Ionomer Type II (F2); a resin-modified glass-ionomer: Fuji Ionomer Type II LC (LC); two "giomer" materials: Reactmer Paste (RP) and Beautifil (BT); a fluoride-containing resin composite: Unifil F (UF); and a non-fluoride resin composite: AP-X (AP) were used in this study. Each material was filled into a plastic mold, with inner diameter of 9 mm wide x 3 mm high. The specimens were stored in vials filled with 8 ml distilled deionized water for 24 hours at 37°C. The specimens were then removed from the vials and the amount of fluoride released into the water, over the 24-hour period, was measured. The amount of fluoride released was measured by using specific fluoride electrode and an ion-analyzer. These procedures were

repeated at Days 2, 3, 7, 14, and 21. After 21 days, all specimens were exposed to 1000 ppm NaF solution for 5 minutes once a day. This procedure and measurement of fluoride release were continued for 14 days. After 14 days, the specimens were placed in water for 7 days and fluoride release was measured. At the 22nd day (1 day after starting fluoride exposure), there was no difference between the F2 and RP, though there were significant differences between the two GICs and the groups BT and UF. After that day, there were significant differences between GIC and the group RP, BT and UF. All materials showed a decrease in fluoride release 7 days after end of the fluoride immersion period. RP and BT revealed lower fluoride release 1 day after the end of the fluoride immersion period as compared to Day 21.

**Al-Naimi OT, Itota T, Hobson RS, McCabe JF (2008)<sup>28</sup>** conducted a study to evaluate the fluoride release for restorative materials and its effect on biofilm formation in natural saliva. Columnar specimens of glass ionomer cement (GIC), resin modified glass ionomer cement (RMGIC), compomer, giomer and composite, were prepared, matured for 24 h at 37°C and 100% humidity, lapped and then placed in natural stimulated saliva with a pH of 3.8 or 7.1. Fluoride release was determined daily using an ion-selective electrode. The surfaces of selected specimens were observed using Confocal Laser Scanning Microscopy in conjunction with a fluorescent dye. The surface biofilm formation and bacterial

growth was most dominant under neutral conditions and on the surfaces of GICs compared with other materials. GICs released significantly higher amounts of fluoride than other materials. The results suggest that the increased fluoride release of GICs did not reduce the amount of bacterial growth and biofilm formation on the surfaces of these materials when stored in natural saliva.

**Mousavinasab S.M, Ian Meyers (2009)<sup>4</sup>** conducted a study to measure the amounts of fluoride released from fluoride-containing materials. Four glass ionomer cements (Fuji IX, Fuji VII, Fuji IX Extra and Fuji II LC), a compomer (Dyract Extra) and a giomer (Beautifil) were used for the study. Twenty cylindrical specimens were prepared from each material. The amount of released fluoride was measured during the first week and on the days 14 and 21 by using specific fluoride electrode and an ion-analyzer. The results were statistically analyzed using analysis of variance (two way ANOVA) and Tukey Kramer multiple comparison tests ( $P=0.05$ ). Significant differences were seen in fluoride release of different days and materials ( $P<0.05$ ). The maximum cumulative fluoride release of days 1-7 was related to Fuji VII, followed by Fuji IX Extra, Fuji II LC, Fuji IX, Dyract Extra and Beautifil in descending order and this order remained the same until the 21st day. An explanation for the high difference in fluoride release between GIC and resin composite like (compomers and giomers) is that, the porosity of the materials may have a great influence on the amounts of fluoride release. Also, these materials have added resin contents compared to

GICs, the barrier through which water and fluoride to diffuse also increases, in addition to their filler solubility differences.

**Trachtenberg F, Maserejian N, Soncini JA, Hayes C, Tavares M (2009)<sup>29</sup>** tested the hypothesis that compomer is associated with fewer future caries compared with amalgam. The five-year trial recruited 534 children aged 6-10 years with  $\geq 2$  carious posterior teeth. Children were randomized to receive compomer or amalgam restorations in primary posterior teeth, placed with a fluoride-releasing bonding agent. The association between restorative material and future caries was assessed by survival analysis. Average follow-up of restorations (N = 1085 compomer, 954 amalgams) was  $2.8 \pm 1.4$  years in 441 children. No significant difference between materials was found in the rate of new caries on different surfaces of the same tooth. Incident caries on other teeth appeared slightly more quickly after placement of compomer restorations ( $P = 0.007$ ), but the difference was negligible after 5 years. Under the conditions of this trial, the authors found no preventive benefit to fluoride-releasing compomer compared with amalgam.

**Dhull KS, Nandlal B (2011)<sup>3</sup>** conducted an in-vitro study to determine the effect of low-concentration daily topical fluoride application on fluoride release of Giomer and Compomer and to compare the amount of fluoride release from Giomer to that of Compomer. Forty-eight specimens of each Giomer and

Compomer were divided into four treatment groups, namely, control group, fluoridated dentifrice (500 ppm) once-daily group, fluoridated dentifrice (500 ppm) twice-daily group and fluoridated dentifrice (500 ppm) once-daily + fluoridated mouthwash (225 ppm) group. Each specimen was suspended in demineralizing solution for 6 h and remineralizing solution for 18 h. Fluoride release was measured in both the demineralizing solution and the remineralizing solution daily for 21 days. The fluoride release (ppm) was found to be higher in Giomer when compared with Compomer. The fluoride released from Giomer and Compomer was significantly higher in the acidic demineralizing solution than in the neutral remineralizing solution. It was found that increasing fluoride exposure significantly increased fluoride release from Giomer and Compomer. It was found that the fluoride release from the subgroups of Giomer and Compomer was in the following order: fluoridated dentifrice twice-daily > fluoridated dentifrice once-daily + fluoridated mouthwash > fluoridated dentifrice once-daily > control group. It was found that Giomer showed a greater fluoride uptake Compomer.

**Mitra SB, Oxman JD, Falsafi A, Ton TT (2011)**<sup>30</sup> conducted a study titled ‘Fluoride release and recharge behavior of a nano-filled resin-modified glass ionomer compared with that of other fluoride releasing materials.’ The study aimed at comparing the long-term fluoride release kinetics of a novel nano-filled two-paste resin-modified glass-ionomer (RMGI), Ketac Nano (KN) with that of two powder-liquid resin-modified glass-ionomers, Fuji II LC (FLC) and Vitremer

(VT) and one conventional glass-ionomer, Fuji IX (FIX). Fluoride release was measured in vitro using ion-selective electrodes. Kinetic analysis was done using regression analysis and compared with existing models for GIs and compomers. In a separate experiment the samples of KN and two conventional glass-ionomers, FIX and Ketac Molar (KM) were subjected to a treatment with external fluoride source (Oral-B Neutra-Foam) after 3 months of fluoride release and the recharge behavior studied for an additional 7-day period. The cumulative amount of fluoride released from KN, VT and FLC and the release profiles were statistically similar but greater than that for FIX at  $P < 0.05$ . All four materials, including KN, showed a burst of fluoride ions at shorter times ( $t$ ) and an overall rate dependence on  $t^{1/2}$  typical for glass-ionomers. The coating of KN with its primer and of DY with its adhesive did not significantly alter the fluoride release behavior of the respective materials. The overall rate for KN was significantly higher than for the compomer DY. DY showed a linear rate of release vs.  $t$  and no burst effect as expected for compomers. The nanoionomer KN showed fluoride recharge behavior similar to the conventional glass ionomers FIX and KM. Thus, it was concluded that the new RMGI KN exhibits fluoride ion release behavior similar to typical conventional and RMGIs and that the primer does not impede the release of fluoride.

**Neelakantan P, John S, Anand S, Sureshababu N, Subbarao C (2011)<sup>31</sup>**

compared the amount and pattern of fluoride release from a new glass-ionomer-



based material (nano-ionomer) with other restorative materials and correlated the surface area to volume of nano-sized filler with its capacity to release fluoride in the powder, more quickly increasing the fluoride. The materials evaluated were a nano-ionomer (Ketac N 100), a conventional glass-ionomer cement (GC Fuji II), a resin-modified glass ionomer cement (GC Fuji II LC), a compomer (Dyract F) and a fluoride-releasing resin composite (Tetric N Flow). A resin composite (Synergy Flow) served as the control. Ten specimens were fabricated from each of these materials using a customized metal mold. The fluoride release was measured every 24 hours for the first seven days, and on days 14, 21 and 28, a combination fluoride ion-selective electrode connected to an ion analyzer. The data was analyzed by one-way ANOVA and Tukey HSD test ( $p=0.05$ ). An initial fluoride “burst effect” was seen with all of the materials, except for the control and compomer. The conventional glass-ionomer cement showed the highest fluoride release on the first three days. The nano-ionomer showed the maximum release of fluoride for the remaining days. A low constant level of fluoride release was seen from the compomer and fluoride-releasing resin composite throughout the study period.

**Paschoal M.A, Gurgel C.V, Daniela Rios, Magalhães A.C, Buzalaf M.A, Machado M.A (2011)<sup>32</sup>** conducted a study to determine the fluoride release profile of a nanofilled resin-modified glass ionomer cement. The aim of the study was to compare the fluoride (F-) release pattern of a nanofilled resin-modified

glass ionomer cement (GIC) (Ketac N100 - KN) with available GICs used in dental practice (resin-modified GIC - Vitremer - V; conventional GIC - Ketac Molar - KM) and a nanofilled resin composite (Filtek Supreme - RC). Discs of each material (n=6) were placed into 4 mL of deionized water in sealed polyethylene vials and shaken, for 15 days. Fluoride release ( $\mu\text{g F}^-/\text{cm}^2$ ) was measured each day using a fluoride-ion specific electrode. Cumulative F<sup>-</sup> release means were statistically analyzed by linear regression analysis. In order to analyze the differences among materials and the influence of time in the daily F<sup>-</sup> release, 2-way ANOVA test was performed ( $\alpha=0.05$ ). The results showed that KN and V presented a strong relationship between cumulative fluoride release and time, which, in turn, means that these materials were able to keep constant fluoride release pattern overtime. Generally, all GICs presented the highest fluoride amount released at the first day. Only for GIC materials (KM, KN and V), there were significant differences between the daily fluoride release overtime up to the third day, after which a plateau was shown. The daily fluoride release means for RC was low and similar overtime. When GICs were compared, KN and V significantly differ from KM up to the seventh and sixth day, respectively; KN was significant different from V up to second day. The authors were able to conclude that the fluoride release profile of the nanofilled resin-modified GIC is comparable to the resin-modified GIC.

**Bahadure RN, Pandey RK, Kumar R, Gopal K, Singh RK (2012)<sup>33</sup>**

estimated the fluoride release from various dental restorative materials at different pH. The study comprised of six dental restorative materials, namely Amalgomer CR, Fuji II, Fuji IX, Beautifil II, Dyract extra, and Coltene Synergy. 30 cylindrical samples of each dental restorative material were prepared using a Teflon mold, having a diameter of 5 mm and height of 3 mm. The 30 samples of each dental restorative material were prepared and grouped into five with six samples in each group as per the pH of the solution 4.3, 4.6, 5.0, 5.5, and 6.2. All the samples were subjected to alternate cycling of the demineralizing solution (6 hours) and remineralizing solution (18 hours) for 15 days. Samples in each subgroup were submitted to their respective pH level of demineralizing solution in polypropylene test tubes. Each test tube contained 2 mL of the demineralizing solution. The test tubes containing samples were subjected to constant shaking for 6 hours in shaking table of orbital shaker, placed in incubator at  $37 \pm 1^\circ\text{C}$ . The samples of each subgroup were removed from the test tubes with the help of tweezers, rinsed with distilled water, dried with blotting paper (or tissue paper) and submitted to the next set of test tubes with each tube containing 2 mL of remineralizing solution. These test tubes were subjected to constant shaking for next 18 hours in shaking table of orbital shaker and placed in incubator at  $37 \pm 1^\circ\text{C}$ . The solutions were collected daily, identified and stored in polypropylene containers at  $4^\circ\text{C}$  to measure fluoride release. The procedure was

continued for 15 days. The fluoride release was measured by using fluoride ion specific electrode and digital ion analyzer. Statistical analysis was done using Pearson correlation analysis and simple linear regression analysis. The result showed that the fluoride release rate was significantly higher in first day and reduced after third day to nearly constant level. At pH 4.3, the fluoride release was highest and lowest at pH 6.2. The Amalgomer CR showed the highest fluoride release among all the experimental dental restorative materials.

**Arbabzadeh-Zavareh F, Gibbs T, Meyers IA, Bouzari M, Mortazavi S, Walsh LJ<sup>34</sup> (2012)** conducted a study with the aim to compare the recharge pattern of six glass ionomer cements after exposure to fluoride. Fuji VII, Fuji IX, Riva Pink, Riva Bleach, Ketac Fil and Fuji IX Extra were investigated. The fluoride-containing materials used were tooth paste and mouth wash (Colgate). Specimens of each material (n=15) were immersed separately in deionized water for 59 days. Then the samples of each material were divided into three groups of five each. Two groups were recharged for 2, 20 and 60 minutes daily during three consecutive weekly intervals and then no treatment for one week. The third group was used as control. Fluoride release measurements ( $\mu\text{g}/\text{cm}^2/\text{day}$ ) were made in every 24 h. One-way and repeated measures analysis of variance tests were used. Tooth paste recharged materials showed higher level of recharge. On day 1, the difference of fluoride release from different treatment groups of different materials except for Fuji IX Extra were not significant ( $P>0.05$ ). On days 7 and

14, the differences observed were significant ( $P < 0.05$ ) for all materials except for Fuji VII (tooth paste versus mouth wash) and Trial Fuji IX (mouth wash versus control) and on day 14 for Rvia Pink (mouth wash versus control). On days 21 and 28, the differences observed were significant for all the materials ( $P < 0.05$ ) except for Riva Pink (toothpaste versus mouth wash), Riva Bleach, Ketac Fil and Trial Fuji IX (mouth wash versus control) on day 28. The authors concluded that a time tabled schedule of application of fluoride-containing materials could help to achieve high fluoride release.

## **MATERIALS AND METHODS**

The present *in vitro* study was conducted by the Department of Pedodontics and Preventive Dentistry, Ragas Dental College & Hospital, Chennai in association with the Department of Safety Engineering, Indira Gandhi Centre for Atomic Research, Kalpakkam to evaluate the fluoride release and recharge of pre-reacted glass ionomer composite and nano-ionomeric glass ionomer with daily topical fluoride supplementation.

### **MATERIALS**

The following materials and equipment were used for the present study:

#### **A. Restorative materials**

1. Pre-reacted glass ionomer composite/‘Giomer’ – Beautifil II<sup>TM</sup>  
(Shofu Inc.)
2. Nano-ionomeric glass ionomer Cement – Ketac<sup>TM</sup> N100 (3M ESPE)

#### **B. Armamentarium for specimen preparation**

1. 5 x 2 mm Teflon mould
2. Agate spatula
3. Mixing pad
4. Plastic filling instrument

5. Mylar strips
6. Microscopic glass slides
7. Light curing unit (Dentsply QHL – 75 curing unit)

C. Immersion media

1. Demineralizing solution
2. Remineralizing solution

D. Topical fluoride agents

1. Sodium monofluorophosphate dentrifice (1000 ppm F)
2. Sodium fluoride mouthrinse (225 ppm F)

E. Equipment

1. Incubator
2. Fluoride ion specific electrode (model 96-09, Orion Research Inc)

F. Chemicals

1. TISAB II (Total Ionic Strength Adjustment Buffer)

G. Others

1. Applicator brush
2. 25 ml plastic containers
3. Measurement pipette

## **METHODOLOGY**

A pre-reacted glass ionomer composite and a nano-ionomeric glass ionomer cement were chosen for this study to check for their ability of fluoride release and recharge. Seventy two specimens (36 of each material)

were made and cured according to the manufacturers' recommendations by placing the restorative materials into the teflon mould (5 mm diameter x 2 mm height).

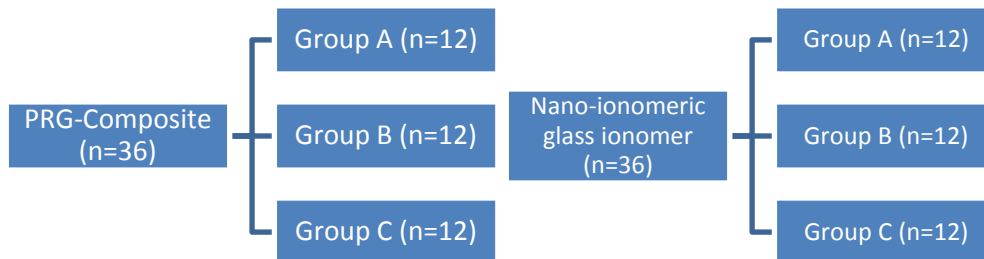
The pre-reacted glass ionomer composite is available in a single dispensing tube. Sufficient material was dispensed onto a paper pad which was then loaded into the teflon mould with the help of a plastic filling instrument. Prior to curing, the material was covered with mylar strips to avoid early moisture contamination. The excess material was removed by placing a glass slide on the upper part of the teflon mould with slight uniform pressure. The material was then cured as per the manufactures recommendation using a Dentsply QHL – 75 curing unit.

The nano-ionomeric glass ionomer is available as a two paste system. Equal amounts of two pastes were dispensed onto a mixing paper pad which was then mixed using an agate spatula for 20 seconds into a uniform viscous mass which in turn was loaded into the teflon mould with the help of a plastic filling instrument. Prior to curing, the material was covered with mylar strips to avoid early moisture contamination. The excess material was removed by placing a glass slide on the upper part of the teflon mould with slight uniform pressure. The nano-ionomeric glass ionomer was then cured as per the manufactures recommendation using a Dentsply QHL – 75 curing unit.

All specimens were stored in deionized water for three days at 37°C to permit complete setting prior to beginning the experimental phase of the study.



Thirty six specimens of each group were subdivided into 3 treatment groups (n=12) as follows:



- Group A - No fluoride treatment (control).
- Group B - Application of a 0.38% w/w sodium monofluorophosphate dentrifice (1000 ppm), for 1 minute, once daily.
- Group C - Same regimen as Group B plus immersion in a 0.05% sodium fluoride mouth rinse (225 ppm F) for one minute immediately following the dentifrice application.

Test specimens were subjected to the daily fluoride exposure protocols according to the groups ascertained. For group A, no fluoride was applied. For group B, application of fluoridated dentrifice (1000 ppm) was done for one minute using an applicator brush, while for group C, after the one minute dentrifice application, the specimens were immersed in a sodium fluoride mouth rinse ( 225 ppm) for 1 minute. After their respective fluoride supplementation, the test specimens were exposed to the pH cycling system.

| Daily Fluoride Exposure Protocol                             |   |                       |
|--|---|-----------------------|
| Sub Groups for Nano-ionomeric glass ionomer & PRG- composite | Fluoride Treatment                                |                       |
|  | Sodium monofluorophosphate dentrifice application | Sodium fluoride rinse |
| A  | Control – No treatment                            |                       |
| B  | ✓   |                       |
| C  | ✓   | ✓                     |

All the specimens were exposed to a pH cycling system (Carvalho & Cury, 1999)<sup>23</sup> consisting of a demineralizing and a remineralizing solution, which was used as the suspension medium for storing the individual specimens. According to Featherstone (1986),<sup>23</sup> this exposure mimics an *in vivo* high caries challenge environment.

The demineralizing solution was prepared by mixing the following constituents at a pH of 4.4.

1. Calcium chloride dehydrate ( $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ ) – 2.2 mM
2. Sodium phosphate dehydrate ( $\text{NaH}_2\text{PO}_4 \cdot 2\text{H}_2\text{O}$ ) – 2.2mM
3. Acetic acid ( $\text{CH}_3\text{COOH}$ ) – 0.05 mM
4. Potassium hydroxide (KOH) – 1 M (to adjust the pH at 4.4)

The remineralization solution was prepared by mixing the following constituents at a pH of 7

1. Calcium chloride dehydrate ( $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ ) – 1 mM
2. Sodium phosphate dehydrate ( $\text{NaH}_2\text{PO}_4 \cdot 2\text{H}_2\text{O}$ ) – 1 mM
3. Sodium chloride ( $\text{NaCl}$ ) – 35 mM
4. Sodium acetate trihydrate ( $\text{CH}_3\text{COONa} \cdot 3\text{H}_2\text{O}$ ) – 15 mM
5. Potassium hydroxide ( $\text{KOH}$ ) – 1 mM (to adjust the pH at 7)

Fluoride treatments were completed prior to the immersion of the samples into the demineralizing solution. Each of the seventy two specimens were stored in a 25 ml plastic container containing 10 ml demineralizing solution at 37°C for 6 hours and then transferred to a new plastic container containing 10 ml remineralization solution for at 37°C for 18 hours. Between the transfers of the samples from the demineralizing solution into the remineralizing solution, the samples were dried by placing them on blotting paper. The temperature was constantly monitored at 37°C by placing the samples in an incubator. The demineralizing and remineralizing solutions were freshly prepared and changed every day.

Fluoride release was determined every day after buffering the demineralizing and remineralizing solutions with equal volumes of TISAB II (Total Ionic Strength Adjustment Buffer). TISAB II is a buffer solution which increases the ionic strength of a solution to a relatively high level. It is used to decomplex fluoride and provide a constant background ionic strength. To prepare the solution, a four liter beaker was half filled with distilled water. The beaker was placed on a magnetic stirrer. 230 ml of concentrated acetic

acid, 232 grams of reagent-grade sodium chloride and 16 grams of reagent grade CDTA (cyclohexylenedinitrilo-tetraacetatic acid) were slowly added. After the solids had been dissolved, the solution was allowed to cool to room temperature, after which 150 grams of reagent grade sodium hydroxide were added. A pH electrode was calibrated and the pH was adjusted to 5.25.

Fluoride content was measured using a fluoride ion specific electrode (Model 96-09, Orion Research Inc, Boston, MA, USA) connected to an Orion ion analyzer (Model EA 940, Orion Research Inc).

For each restorative material/fluoride treatment combination, daily fluoride release (mean  $\pm$  SD) in both demineralization and remineralization solutions were calculated. Total daily fluoride release was calculated by adding the amount released in the demineralizing solution to that released in the remineralizing solution. For each material, the ability to “recharge” was calculated as the difference in fluoride release between the treatment groups and the control group.<sup>3</sup>

The data from the experimental procedure was tabulated and statistically analyzed. Statistical analysis was done using repeated measures ANOVA, one way ANOVA followed by post hoc Tukey test and unpaired ‘t’ test.

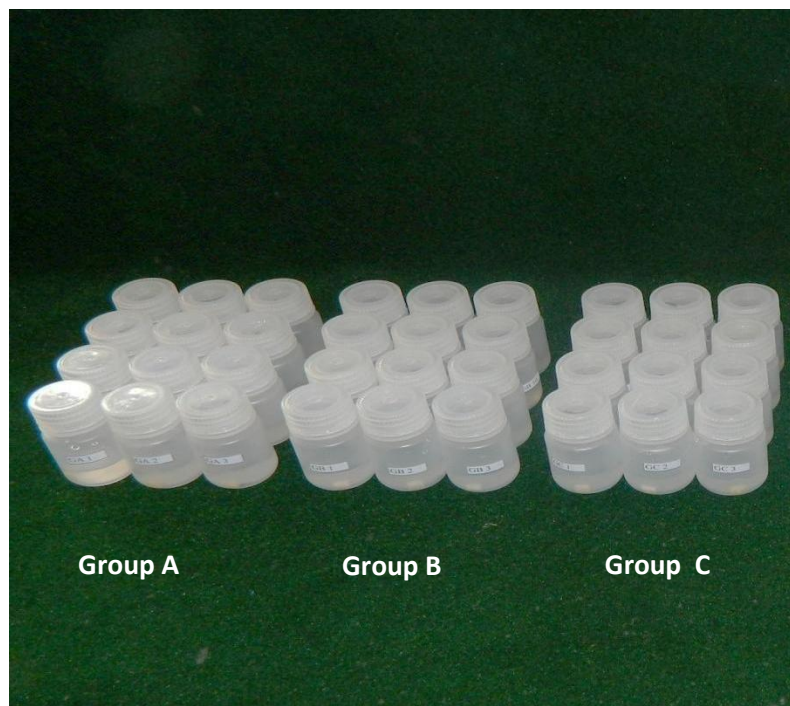
### FIGURE 1: ARMAMENTARIUM



**FIGURE 2: PRE-REACTED GLASS IONOMER COMPOSITE SPECIMENS IN DEMINERALIZING SOLUTION**

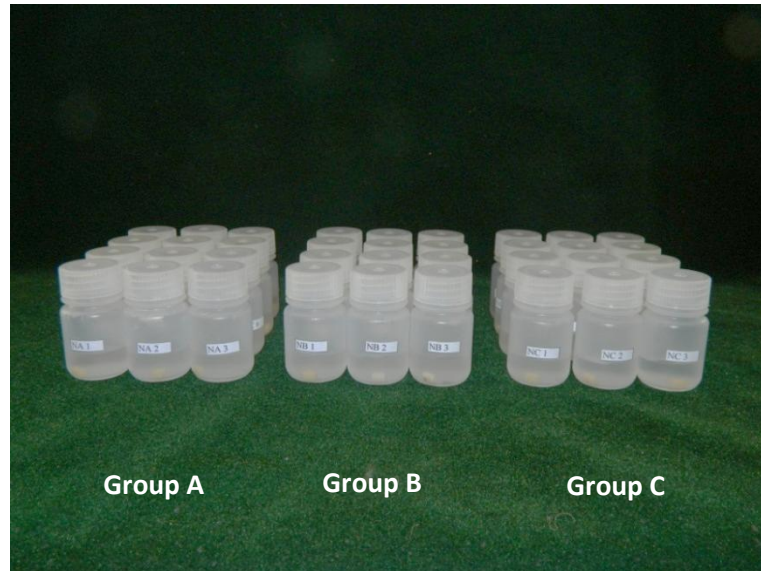


**FIGURE 3: PRE-REACTED GLASS IONOMER COMPOSITE SPECIMENS IN REMINERALIZING SOLUTION**

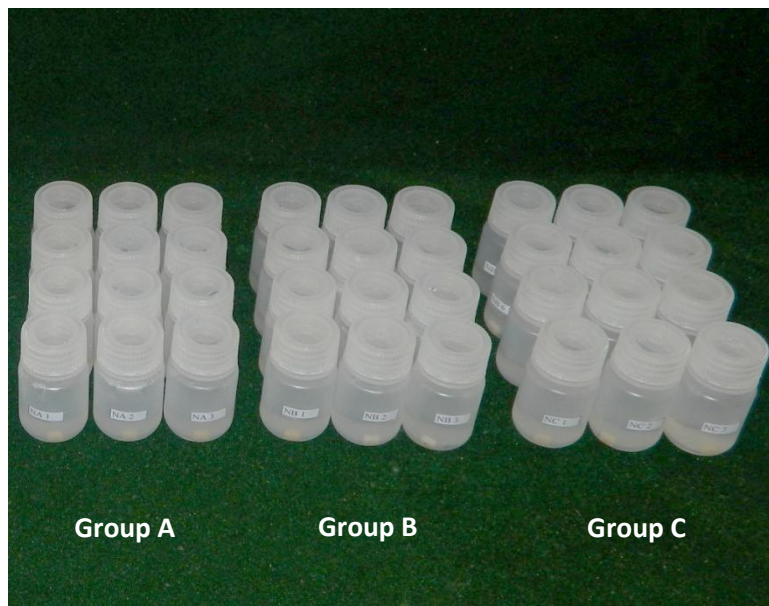




**FIGURE 4: NANO-IONOMERIC GLASS IONOMERIC SPECIMENS  
IN DEMINERALIZING SOLUTIONS**



**FIGURE 5: NANO-IONOMERIC GLASS IONOMERIC SPECIMENS  
IN REMINERALIZING SOLUTIONS**



**FIGURE 6: INCUBATOR**



**FIGURE 7: SAMPLES INSIDE THE INCUBATOR**





**FIGURE 8: FLUORIDE ION SPECIFIC ELECTRODE**



## RESULTS

Seventy two specimens (thirty six of each restorative material) of pre-reacted glass ionomer composite and nano – ionomeric glass ionomer were analyzed for release and recharge of fluoride with daily fluoride supplementation using fluoride ion specific electrode. The data obtained were tabulated and statistically analyzed with repeated measures ANOVA, one way ANOVA followed by post hoc Tukey test and unpaired ‘t’ test using SPSS software (version 17.0). A P value of < 0.05 was considered to be significant.

**Table 1** represents the mean fluoride release from pre-reacted glass ionomer composite into demineralizing and remineralizing solutions with different fluoride treatment protocols for a period of 21 days. The fluoride release from pre-reacted glass ionomer composite into the demineralizing solution for the control group (without fluoride treatment) on day 1 was  $3.994 \pm 0.07$  ppm which was reduced to  $0.28 \pm 0.05$  ppm on day 21. With single fluoride application, fluoride release ranged from  $4.198 \pm 0.09$  ppm on day 1 to  $0.377 \pm 0.09$  ppm on day 21, whereas with the addition of fluoride mouth rinse along with the fluoride dentrifice, the fluoride release ranged from  $4.329 \pm 0.09$  ppm on day 1 to  $0.449 \pm 0.05$  on day 21. In the remineralizing solution the fluoride release of pre-reacted glass ionomer composite in the control group ranged from  $1.378 \pm 0.09$  ppm on day 1 to  $0.176 \pm 0.04$  ppm on day 21. With single fluoride application, fluoride release into the remineralizing solution ranged from  $1.498 \pm 0.04$  ppm on day 1 which reduced to  $0.241 \pm 0.02$  ppm on

day 21, whereas with the addition of fluoride mouth rinse along with the fluoride dentrifice, the fluoride release ranged from  $1.673 \pm 0.07$  ppm on day 1 to  $0.327 \pm 0.08$  ppm on day 21. The results showed that fluoride release and recharge was influenced by immersion media for pre-reacted glass ionomer composite. Regardless of the fluoride treatment, the mean fluoride release for pre-reacted glass ionomer composite was significantly greater when immersed in the demineralizing solution than in the remineralizing solution, even though the specimens were suspended in the remineralizing solution for thrice the duration when compared to specimens suspended in the demineralizing solution.

**Table 2** represents the mean fluoride release from nano – ionomeric glass ionomer into demineralizing and remineralizing solutions with different fluoride treatment protocols for a period of 21 days. The fluoride release from nano – ionomeric glass ionomer into the demineralizing solution for the control group (without fluoride treatment) on day 1 was  $5.294 \pm 0.08$  ppm which was reduced to  $0.66 \pm 0.04$  ppm on day 21. With single fluoride application, fluoride release ranged from  $5.698 \pm 0.06$  ppm on day 1 to  $0.844 \pm 0.04$  ppm on day 21, whereas with the addition of fluoride mouth rinse along with the fluoride dentrifice, the fluoride release ranged from  $5.843 \pm 0.09$  ppm on day 1 to  $0.926 \pm 0.05$  ppm on day 21. In the remineralizing solution the fluoride release of nano – ionomeric glass ionomer in the control group ranged from  $2.542 \pm 0.07$  ppm on day 1 to  $0.487 \pm 0.05$  ppm on day 21. With single

fluoride application, fluoride release into the remineralizing solution ranged from  $2.745 \pm 0.09$  ppm on day 1 which then reduced to  $0.641 \pm 0.04$  ppm on day 21, whereas with the addition of fluoride mouth rinse along with the fluoride dentrifice, the fluoride release ranged from  $2.876 \pm 0.08$  ppm on day 1 to  $0.756 \pm 0.08$  ppm on day 21. The results showed that fluoride release and recharge was influenced by immersion media for nano – ionomeric glass ionomer. Regardless of the fluoride treatment, the mean fluoride release for nano – ionomeric glass ionomer was significantly greater when immersed in the demineralizing solution than in the remineralizing solution, even though the specimens were suspended in the remineralizing solution for thrice the duration when compared to specimens suspended in the demineralizing solution.

**Table 3** shows the comparative evaluation of fluoride release from pre-reacted glass ionomer and nano-ionomeric glass ionomer into demineralizing and remineralizing solutions at weekly intervals for a period of 21 days with daily fluoride supplements. Immersion media played an important role in the amount of fluoride released for both the materials. There was a statistically significant difference between the amount of fluoride released into the demineralizing and remineralizing solutions ( $P=0.000^{**}$ ) for both the restorative materials, with more amount of fluoride being released into the demineralizing solution for all the fluoride treatment groups.

On comparing the amount of fluoride released over a period of time, repeated measures ANOVA showed significant differences in fluoride release between days 1, 7, 14 and 21 ( $P = 0.000^{**}$ ) for both materials in demineralizing and remineralizing solutions for all fluoride treatment protocols.

**Table 4 and graph 1** shows the comparative evaluation of the effect of daily fluoride supplement on fluoride release from pre-reacted glass ionomer composite and nano-ionomeric glass ionomer. The fluoride release was calculated by adding fluoride that was released into demineralizing solution with that released into the remineralizing solution on each day from days 1 to 7, and on days 14 and 21. Fluoride release was significantly greater with the fluoride dentrifice and combined fluoride application, when compared to the control group for both the restorative materials. The combination group had a significantly greater fluoride release for both the materials than the group in which dentrifice alone was applied ( $P < 0.010$ ). The two restorative materials demonstrated a decrease in the amount of fluoride released over the course of the study, with nano-ionomeric glass ionomer having showed a higher amount of fluoride release than pre-reacted glass ionomer composite. Fluoride release was related to the dose of daily supplemental fluoride which was applied. There was an initial rapid release of high amount of fluoride within the first two days by both the restorative materials. This initial rapid release had been termed as “fluoride burst”.

In group C, the pre-reacted glass ionomer composite showed gradual decrease in the amount of fluoride released from day 1 ( $6.022 \pm 0.14$  ppm) until day 7 ( $3.061 \pm 0.10$  ppm) and from then onwards, a drastic decrease to the minimum amount by day 21 ( $0.776 \pm 0.15$  ppm) was seen (**Graph 1**). In group C, the fluoride release from the nano-ionomeric glass ionomer decreased gradually from day 1 ( $8.713 \pm 0.14$  ppm) until day 3 ( $7.091 \pm 0.08$  ppm), followed by a sustained release of fluoride with slightest decrease in the amount of fluoride release from day 4 ( $5.8 \pm 0.14$  ppm) until day 7 ( $5.046 \pm 0.09$  ppm) which was followed by a drastic decrease in the amount of fluoride released to the minimum on day 21 ( $1.682 \pm 0.11$  ppm).

Similar type of fluoride release pattern was observed in other fluoride treatment protocols (Groups B, A) for both pre-reacted glass ionomer composite ( $5.696 \pm 0.08$  -  $2.844 \pm 0.08$  -  $0.618 \pm 0.07$ ,  $5.372 \pm 0.29$  -  $2.641 \pm 0.14$  -  $0.456 \pm 0.12$ ) and nano-ionomeric glass ionomer ( $8.443 \pm 0.12$  -  $6.846 \pm 0.15$  -  $5.6 \pm 0.08$  -  $4.796 \pm 0.12$  -  $1.485 \pm 0.14$ ,  $7.837 \pm 0.13$  -  $6.505 \pm 0.12$  -  $5.306 \pm 0.13$  -  $4.476 \pm 0.07$  -  $1.147 \pm 0.08$ ).

**Table 5** shows the comparison of mean fluoride release (mean  $\pm$  S.D) between pre-reacted glass ionomer composite and nano-ionomeric glass ionomer on days 1,7,14 and 21. There was a significant difference in the amount of fluoride released between nano-ionomeric glass ionomer and pre-reacted glass ionomer composite ( $P = 0.000^{**}$ ). There was a higher fluoride release from nano-ionomeric glass ionomer as compared with pre-reacted

glass ionomer composite, irrespective of the fluoride treatment protocol throughout the study period.

**Table 6 (Graph 2 and Graph 3)** shows the recharging ability of pre-reacted glass ionomer composite and nano-ionomeric glass ionomer. The recharge ability was calculated as the difference in the fluoride release between treatment groups and the control group.<sup>3</sup> No sustained pattern of recharge was shown in both the groups.

**Graph 2** The pre-reacted glass ionomer composite group, showed maximum recharge on day 1 and the least on day 21, whereas for the nano-ionomeric glass ionomer, maximum recharge was seen on day 2 and the least was on day 5. (Control group v/s group C)

**Graph 3** The pre-reacted glass ionomer composite group showed maximum recharge on day 1 and the least on day 3, whereas for the nano-ionomeric glass ionomer group, maximum recharge was seen on day 2 and the least on day 6. (Control group v/s group B)

When compared between both the materials, nano-ionomeric glass ionomer showed greater rechargability than pre-reacted glass ionomer composite

**Table 1: Daily Fluoride Release (Mean  $\pm$  S.D) (Ppm) From Pre-Reacted Glass Ionomer Composite into Demineralization and Remineralization Solutions with Different Fluoride Treatment Protocols**

| DAYS | Demineralizing solution |                  |                  | Remineralizing solution |                  |                  |
|------|-------------------------|------------------|------------------|-------------------------|------------------|------------------|
|      | Group A                 | Group B          | Group C          | Group A                 | Group B          | Group C          |
| 1    | 3.994 $\pm$ 0.07        | 4.198 $\pm$ 0.09 | 4.329 $\pm$ 0.09 | 1.378 $\pm$ 0.09        | 1.498 $\pm$ 0.04 | 1.673 $\pm$ 0.07 |
| 2    | 3.687 $\pm$ 0.07        | 3.759 $\pm$ 0.09 | 3.865 $\pm$ 0.07 | 1.296 $\pm$ 0.03        | 1.376 $\pm$ 0.07 | 1.598 $\pm$ 0.08 |
| 3    | 3.33 $\pm$ 0.05         | 3.327 $\pm$ 0.08 | 3.518 $\pm$ 0.06 | 1.179 $\pm$ 0.05        | 1.267 $\pm$ 0.07 | 1.521 $\pm$ 0.06 |
| 4    | 3.094 $\pm$ 0.09        | 3.117 $\pm$ 0.06 | 3.294 $\pm$ 0.07 | 1.065 $\pm$ 0.05        | 1.189 $\pm$ 0.09 | 1.489 $\pm$ 0.09 |
| 5    | 2.686 $\pm$ 0.09        | 2.765 $\pm$ 0.07 | 2.957 $\pm$ 0.09 | 1.012 $\pm$ 0.06        | 1.138 $\pm$ 0.09 | 1.395 $\pm$ 0.06 |
| 6    | 2.287 $\pm$ 0.05        | 2.358 $\pm$ 0.05 | 2.548 $\pm$ 0.05 | 0.987 $\pm$ 0.05        | 1.024 $\pm$ 0.07 | 1.21 $\pm$ 0.02  |
| 7    | 1.705 $\pm$ 0.04        | 1.844 $\pm$ 0.05 | 2.018 $\pm$ 0.05 | 0.936 $\pm$ 0.04        | 1 $\pm$ 0.03     | 1.043 $\pm$ 0.04 |
| 8    | 1.602 $\pm$ 0.07        | 1.694 $\pm$ 0.06 | 1.831 $\pm$ 0.08 | 0.894 $\pm$ 0.07        | 0.984 $\pm$ 0.05 | 1.005 $\pm$ 0.07 |
| 9    | 1.119 $\pm$ 0.06        | 1.278 $\pm$ 0.05 | 1.587 $\pm$ 0.05 | 0.835 $\pm$ 0.06        | 0.901 $\pm$ 0.09 | 0.985 $\pm$ 0.07 |
| 10   | 0.945 $\pm$ 0.05        | 1.109 $\pm$ 0.07 | 1.265 $\pm$ 0.04 | 0.793 $\pm$ 0.09        | 0.863 $\pm$ 0.08 | 0.954 $\pm$ 0.05 |
| 11   | 0.917 $\pm$ 0.03        | 1.05 $\pm$ 0.08  | 1.174 $\pm$ 0.08 | 0.738 $\pm$ 0.07        | 0.746 $\pm$ 0.06 | 0.832 $\pm$ 0.06 |
| 12   | 0.888 $\pm$ 0.07        | 0.934 $\pm$ 0.04 | 1.079 $\pm$ 0.04 | 0.717 $\pm$ 0.06        | 0.798 $\pm$ 0.05 | 0.856 $\pm$ 0.08 |
| 13   | 0.831 $\pm$ 0.05        | 0.87 $\pm$ 0.03  | 1 $\pm$ 0.06     | 0.576 $\pm$ 0.09        | 0.76 $\pm$ 0.08  | 0.864 $\pm$ 0.04 |
| 14   | 0.748 $\pm$ 0.08        | 0.856 $\pm$ 0.06 | 0.996 $\pm$ 0.06 | 0.508 $\pm$ 0.07        | 0.679 $\pm$ 0.07 | 0.798 $\pm$ 0.09 |
| 15   | 0.712 $\pm$ 0.05        | 0.799 $\pm$ 0.05 | 0.888 $\pm$ 0.07 | 0.467 $\pm$ 0.05        | 0.677 $\pm$ 0.03 | 0.763 $\pm$ 0.08 |
| 16   | 0.665 $\pm$ 0.07        | 0.698 $\pm$ 0.08 | 0.813 $\pm$ 0.06 | 0.431 $\pm$ 0.07        | 0.476 $\pm$ 0.08 | 0.517 $\pm$ 0.06 |
| 17   | 0.617 $\pm$ 0.03        | 0.675 $\pm$ 0.04 | 0.737 $\pm$ 0.07 | 0.396 $\pm$ 0.07        | 0.469 $\pm$ 0.04 | 0.509 $\pm$ 0.03 |
| 18   | 0.561 $\pm$ 0.07        | 0.649 $\pm$ 0.05 | 0.719 $\pm$ 0.03 | 0.372 $\pm$ 0.03        | 0.441 $\pm$ 0.07 | 0.508 $\pm$ 0.04 |
| 19   | 0.47 $\pm$ 0.06         | 0.545 $\pm$ 0.09 | 0.641 $\pm$ 0.04 | 0.315 $\pm$ 0.06        | 0.382 $\pm$ 0.08 | 0.45 $\pm$ 0.05  |
| 20   | 0.378 $\pm$ 0.08        | 0.479 $\pm$ 0.05 | 0.549 $\pm$ 0.07 | 0.259 $\pm$ 0.03        | 0.351 $\pm$ 0.06 | 0.414 $\pm$ 0.04 |
| 21   | 0.28 $\pm$ 0.05         | 0.377 $\pm$ 0.09 | 0.449 $\pm$ 0.05 | 0.176 $\pm$ 0.04        | 0.241 $\pm$ 0.02 | 0.327 $\pm$ 0.08 |

P Value (repeated measures ANOVA): 0.000\*\* (95% confidence interval for mean)

Group A= Control

Group B= Fluoride dentrifice application

Group C= Fluoride dentrifice + rinse application

The results from table showed that fluoride release of pre-reacted glass ionomer composite was influenced by the immersion media. Regardless of the fluoride treatment, the mean fluoride release was significantly greater when the test specimens were immersed in the demineralizing solution than in the remineralizing solution, even though the specimens were suspended in the remineralizing solution for thrice the duration than compared to specimens suspended in the demineralizing solution.



**Table 2: Daily Fluoride Release (Mean  $\pm$  S.D) (ppm) from Nano-Ionomeric Glass Ionomer into Demineralization and Remineralization Solutions with Different Fluoride Treatment Protocols**

| DAYS | Demineralizing solution |                  |                  | Remineralizing solution |                  |                  |
|------|-------------------------|------------------|------------------|-------------------------|------------------|------------------|
|      | Group A                 | Group B          | Group C          | Group A                 | Group B          | Group C          |
| 1    | 5.294 $\pm$ 0.08        | 5.698 $\pm$ 0.06 | 5.843 $\pm$ 0.09 | 2.542 $\pm$ 0.07        | 2.745 $\pm$ 0.09 | 2.876 $\pm$ 0.08 |
| 2    | 4.987 $\pm$ 0.06        | 5.507 $\pm$ 0.07 | 5.534 $\pm$ 0.09 | 2.346 $\pm$ 0.04        | 2.623 $\pm$ 0.09 | 2.742 $\pm$ 0.07 |
| 3    | 4.486 $\pm$ 0.08        | 4.647 $\pm$ 0.09 | 4.872 $\pm$ 0.07 | 2.019 $\pm$ 0.08        | 2.199 $\pm$ 0.08 | 2.219 $\pm$ 0.04 |
| 4    | 3.342 $\pm$ 0.09        | 3.552 $\pm$ 0.09 | 3.643 $\pm$ 0.08 | 1.964 $\pm$ 0.09        | 2.048 $\pm$ 0.04 | 2.157 $\pm$ 0.08 |
| 5    | 3.178 $\pm$ 0.04        | 3.296 $\pm$ 0.01 | 3.418 $\pm$ 0.06 | 1.903 $\pm$ 0.06        | 2.007 $\pm$ 0.03 | 2.045 $\pm$ 0.09 |
| 6    | 2.989 $\pm$ 0.03        | 3.021 $\pm$ 0.02 | 3.276 $\pm$ 0.02 | 1.879 $\pm$ 0.03        | 1.945 $\pm$ 0.03 | 1.998 $\pm$ 0.06 |
| 7    | 2.675 $\pm$ 0.08        | 2.896 $\pm$ 0.06 | 3.103 $\pm$ 0.09 | 1.801 $\pm$ 0.08        | 1.9 $\pm$ 0.04   | 1.943 $\pm$ 0.08 |
| 8    | 2.443 $\pm$ 0.06        | 2.754 $\pm$ 0.03 | 2.997 $\pm$ 0.04 | 1.782 $\pm$ 0.09        | 1.885 $\pm$ 0.07 | 1.912 $\pm$ 0.04 |
| 9    | 2.356 $\pm$ 0.07        | 2.683 $\pm$ 0.08 | 2.848 $\pm$ 0.03 | 1.736 $\pm$ 0.04        | 1.857 $\pm$ 0.06 | 1.9 $\pm$ 0.09   |
| 10   | 2.198 $\pm$ 0.04        | 2.288 $\pm$ 0.07 | 2.453 $\pm$ 0.07 | 1.662 $\pm$ 0.08        | 1.834 $\pm$ 0.07 | 1.897 $\pm$ 0.05 |
| 11   | 1.792 $\pm$ 0.05        | 2.011 $\pm$ 0.06 | 2.192 $\pm$ 0.05 | 1.528 $\pm$ 0.04        | 1.74 $\pm$ 0.08  | 1.884 $\pm$ 0.06 |
| 12   | 1.659 $\pm$ 0.05        | 1.837 $\pm$ 0.06 | 1.928 $\pm$ 0.04 | 1.403 $\pm$ 0.08        | 1.52 $\pm$ 0.05  | 1.604 $\pm$ 0.07 |
| 13   | 1.524 $\pm$ 0.06        | 1.67 $\pm$ 0.03  | 1.772 $\pm$ 0.05 | 1.292 $\pm$ 0.05        | 1.497 $\pm$ 0.08 | 1.626 $\pm$ 0.04 |
| 14   | 1.419 $\pm$ 0.07        | 1.604 $\pm$ 0.05 | 1.707 $\pm$ 0.05 | 1.19 $\pm$ 0.07         | 1.352 $\pm$ 0.07 | 1.474 $\pm$ 0.07 |
| 15   | 1.303 $\pm$ 0.04        | 1.512 $\pm$ 0.06 | 1.609 $\pm$ 0.04 | 1.101 $\pm$ 0.06        | 1.318 $\pm$ 0.07 | 1.436 $\pm$ 0.03 |
| 16   | 1.156 $\pm$ 0.05        | 1.417 $\pm$ 0.07 | 1.49 $\pm$ 0.05  | 0.974 $\pm$ 0.05        | 1.275 $\pm$ 0.07 | 1.375 $\pm$ 0.07 |
| 17   | 1.008 $\pm$ 0.06        | 1.233 $\pm$ 0.04 | 1.358 $\pm$ 0.07 | 0.863 $\pm$ 0.04        | 1.045 $\pm$ 0.05 | 1.172 $\pm$ 0.04 |
| 18   | 0.953 $\pm$ 0.05        | 1.166 $\pm$ 0.06 | 1.252 $\pm$ 0.06 | 0.77 $\pm$ 0.04         | 0.959 $\pm$ 0.08 | 1.085 $\pm$ 0.03 |
| 19   | 0.876 $\pm$ 0.07        | 0.953 $\pm$ 0.05 | 1.156 $\pm$ 0.04 | 0.684 $\pm$ 0.08        | 0.77 $\pm$ 0.04  | 0.864 $\pm$ 0.05 |
| 20   | 0.767 $\pm$ 0.06        | 0.926 $\pm$ 0.03 | 1.134 $\pm$ 0.05 | 0.59 $\pm$ 0.07         | 0.766 $\pm$ 0.06 | 0.862 $\pm$ 0.04 |
| 21   | 0.66 $\pm$ 0.04         | 0.844 $\pm$ 0.04 | 0.926 $\pm$ 0.05 | 0.487 $\pm$ 0.05        | 0.641 $\pm$ 0.04 | 0.756 $\pm$ 0.08 |

P Value (repeated measures ANOVA): 0.000\*\*

(95% confidence interval for mean)

Group A= Control

Group B= Fluoride dentrifice application

Group C= Fluoride dentrifice + rinse application

The results showed that fluoride release of nano-ionomeric glass ionomer was influenced by immersion media. Regardless of the fluoride treatment, the mean fluoride release was significantly greater when test specimens were immersed in the demineralizing solution than in the remineralizing solution, even though the specimens were suspended in the remineralizing solution for thrice the duration than compared to specimens suspended in the demineralizing solution.

**Table 3: Comparative Evaluation of Fluoride Release from Pre-Reacted Glass Ionomer and Nano-Ionomeric Glass Ionomer Into Demineralizing and Remineralizing Solutions at Weekly Intervals for A Period of 21 Days With Daily Fluoride Supplements**

| Materials                      | Treatment Protocols               | Immersion media | Day 1      | Day 7      | Day 14     | Day 21     | P value |
|--------------------------------|-----------------------------------|-----------------|------------|------------|------------|------------|---------|
| PRG - composite                | Group A (no fluoride application) | Demin           | 3.994±0.07 | 1.705±0.04 | 0.748±0.08 | 0.28 ±0.05 | 0.000** |
|                                |                                   | Remin           | 1.378±0.09 | 0.936±0.04 | 0.508±0.07 | 0.176±0.04 | 0.000** |
|                                | P value                           |                 | 0.000**    | 0.000**    | 0.000**    | 0.000**    |         |
|                                | Group B (dentrifice)              | Demin           | 4.198±0.09 | 1.844±0.05 | 0.856±0.06 | 0.377±0.09 | 0.000** |
|                                |                                   | Remin           | 1.498±0.04 | 1±0.03     | 0.679±0.07 | 0.241±0.02 | 0.000** |
|                                | P value                           |                 | 0.000**    | 0.000**    | 0.000**    | 0.000**    |         |
|                                | Group C ( dentrifice + rinse )    | Demin           | 4.329±0.09 | 2.018±0.05 | 0.996±0.06 | 0.449±0.05 | 0.000** |
|                                |                                   | Remin           | 1.673±0.07 | 1.043±0.04 | 0.798±0.09 | 0.327±0.08 | 0.000** |
|                                | P value                           |                 | 0.000**    | 0.000**    | 0.000**    | 0.004**    |         |
| Nano – ionomeric glass ionomer | Group A (no fluoride application) | Demin           | 5.294±0.08 | 2.675±0.08 | 1.419±0.07 | 0.66±0.04  | 0.000** |
|                                |                                   | Remin           | 2.542±0.07 | 1.801±0.08 | 1.19±0.07  | 0.487±0.05 | 0.000** |
|                                | P value                           |                 | 0.000**    | 0.000**    | 0.000**    | 0.000**    |         |
|                                | Group B (dentrifice)              | Demin           | 5.698±0.06 | 2.896±0.06 | 1.604±0.05 | 0.844±.04  | 0.000** |
|                                |                                   | Remin           | 2.745±0.09 | 1.9±0.04   | 1.352±0.07 | 0.641±0.04 | 0.000** |
|                                | P value                           |                 | 0.000**    | 0.000**    | 0.000**    | 0.000**    |         |
|                                | Group C ( dentrifice + rinse )    | Demin           | 5.843±0.09 | 3.103±0.09 | 1.707±0.05 | 0.926±.05  | 0.000** |
|                                |                                   | Remin           | 2.876±0.08 | 1.943±0.08 | 1.474±0.07 | 0.756±0.08 | 0.000** |
|                                | P value                           |                 | 0.000**    | 0.000**    | 0.000**    | 0.000**    |         |

P Value (repeated measures ANOVA): 0.000\*\*

P Value:

0.000 to 0.010 – significant at 1 %(\*\*)

0.01 to 0.050 – significant at 5 % (\*)

> 0.05 – not significant at 5 %

There was a statistically significant difference between the amount of fluoride released into the demineralizing and remineralizing solutions (P=0.000\*\*) for both the restorative materials with more amount of fluoride being released into the demineralizing solution. On comparing the amount of fluoride released over a period of time, repeated measures ANOVA showed significant differences in fluoride release from day 1 to day 21 (P=0.000\*\*) for both the materials in demineralizing and remineralizing solutions.

**Table 4: Comparative Evaluation of the Effect of Daily Fluoride Supplement on Fluoride Release (Mean  $\pm$  S.D) from Pre-Reacted Glass Ionomer Composite and Nano-Ionomeric Glass Ionomer**

| Material                     | Treatment Groups | Day 1                         | Day 2                         | Day 3                         | Day 4                         | Day 5                         | Day 6                         | Day 7                         | Day 14                        | Day 21                        |
|------------------------------|------------------|-------------------------------|-------------------------------|-------------------------------|-------------------------------|-------------------------------|-------------------------------|-------------------------------|-------------------------------|-------------------------------|
| PRG composite                | Group C          | 6.002 $\pm$ 0.14 <sup>A</sup> | 5.463 $\pm$ 0.11 <sup>A</sup> | 5.039 $\pm$ 0.08 <sup>A</sup> | 4.783 $\pm$ 0.10 <sup>A</sup> | 4.352 $\pm$ 0.10 <sup>A</sup> | 3.758 $\pm$ 0.12 <sup>A</sup> | 3.061 $\pm$ 0.10 <sup>A</sup> | 1.794 $\pm$ 0.08 <sup>A</sup> | 0.776 $\pm$ 0.15 <sup>A</sup> |
|                              | Group B          | 5.696 $\pm$ 0.08 <sup>B</sup> | 5.135 $\pm$ 0.14 <sup>B</sup> | 4.594 $\pm$ 0.11 <sup>B</sup> | 4.306 $\pm$ 0.14 <sup>B</sup> | 3.903 $\pm$ 0.11 <sup>B</sup> | 3.382 $\pm$ 0.10 <sup>B</sup> | 2.844 $\pm$ 0.08 <sup>B</sup> | 1.535 $\pm$ 0.11 <sup>B</sup> | 0.618 $\pm$ 0.07 <sup>B</sup> |
|                              | Group A          | 5.372 $\pm$ 0.29 <sup>C</sup> | 4.983 $\pm$ 0.08 <sup>C</sup> | 4.509 $\pm$ 0.07 <sup>C</sup> | 4.159 $\pm$ 0.11 <sup>C</sup> | 3.698 $\pm$ 0.09 <sup>C</sup> | 3.274 $\pm$ 0.08 <sup>C</sup> | 2.641 $\pm$ 0.14 <sup>C</sup> | 1.256 $\pm$ 0.14 <sup>C</sup> | 0.456 $\pm$ 0.12 <sup>C</sup> |
|                              | P value          | 0.000**                       | 0.000**                       | 0.000**                       | 0.000**                       | 0.000**                       | 0.000**                       | 0.000**                       | 0.000**                       | 0.000**                       |
| Nano-ionomeric glass ionomer | Group C          | 8.713 $\pm$ 0.14 <sup>A</sup> | 8.276 $\pm$ 0.10 <sup>A</sup> | 7.091 $\pm$ 0.08 <sup>A</sup> | 5.800 $\pm$ 0.14 <sup>A</sup> | 5.463 $\pm$ 0.12 <sup>A</sup> | 5.274 $\pm$ 0.15 <sup>A</sup> | 5.046 $\pm$ 0.09 <sup>A</sup> | 3.181 $\pm$ 0.09 <sup>A</sup> | 1.682 $\pm$ 0.11 <sup>A</sup> |
|                              | Group B          | 8.443 $\pm$ 0.12 <sup>B</sup> | 8.130 $\pm$ 0.09 <sup>B</sup> | 6.846 $\pm$ 0.15 <sup>B</sup> | 5.600 $\pm$ 0.08 <sup>B</sup> | 5.303 $\pm$ 0.09 <sup>B</sup> | 4.966 $\pm$ 0.07 <sup>B</sup> | 4.796 $\pm$ 0.12 <sup>B</sup> | 2.956 $\pm$ 0.12 <sup>B</sup> | 1.485 $\pm$ 0.14 <sup>B</sup> |
|                              | Group A          | 7.837 $\pm$ 0.13 <sup>C</sup> | 7.333 $\pm$ 0.06 <sup>C</sup> | 6.505 $\pm$ 0.12 <sup>C</sup> | 5.306 $\pm$ 0.13 <sup>C</sup> | 5.081 $\pm$ 0.06 <sup>C</sup> | 4.868 $\pm$ 0.11 <sup>C</sup> | 4.476 $\pm$ 0.07 <sup>C</sup> | 2.609 $\pm$ 0.15 <sup>C</sup> | 1.147 $\pm$ 0.08 <sup>C</sup> |
|                              | P value          | 0.000**                       | 0.000**                       | 0.000**                       | 0.000**                       | 0.000**                       | 0.000**                       | 0.000**                       | 0.000**                       | 0.000**                       |

One way ANOVA followed by post hoc Tukey test and unpaired 't' test.

Within the same column, different alphabets as superscript denote statistically significant difference between two groups

The nano-ionomeric glass ionomer showed better amount of fluoride release than pre-reacted glass ionomer composite throughout the course of the experiment period irrespective of the fluoride treatment supplementation.

**TABLE 5: COMPARISON OF FLUORIDE RELEASE (MEAN  $\pm$ S.D) (PPM) BETWEEN PRE-REACTED GLASS IONOMER COMPOSITE AND NANO-IONOMERIC GLASS IONOMER (DAYS 1,7,14 AND 21)**

| MATERIAL                                     | Day 1            |                  |                  | Day 7            |                  |                  | Day 14           |                  |                  | Day 21           |                  |                  |
|--|------------------|------------------|------------------|------------------|------------------|------------------|------------------|------------------|------------------|------------------|------------------|------------------|
|  | Group A          | Group B          | Group C          | Group A          | Group B          | Group C          | Group A          | Group B          | Group C          | Group A          | Group B          | Group C          |
| <b>PRG - composite</b>                       | 5.372 $\pm$ 0.29 | 5.696 $\pm$ 0.08 | 6.002 $\pm$ 0.14 | 2.641 $\pm$ 0.14 | 2.844 $\pm$ 0.08 | 3.061 $\pm$ 0.10 | 1.256 $\pm$ 0.14 | 1.535 $\pm$ 0.11 | 1.794 $\pm$ 0.08 | 0.456 $\pm$ 0.12 | 0.618 $\pm$ 0.07 | 0.776 $\pm$ 0.15 |
| <b>Nano-<br/>ionomeric<br/>glass ionomer</b> | 7.837 $\pm$ 0.13 | 8.443 $\pm$ 0.12 | 8.713 $\pm$ 0.14 | 4.476 $\pm$ 0.07 | 4.796 $\pm$ 0.12 | 5.046 $\pm$ 0.09 | 2.609 $\pm$ 0.15 | 2.956 $\pm$ 0.12 | 3.181 $\pm$ 0.09 | 1.147 $\pm$ 0.08 | 1.485 $\pm$ 0.14 | 1.682 $\pm$ 0.11 |
| <b>P value</b>                               | 0.000**          | 0.000**          | 0.000**          | 0.000**          | 0.000**          | 0.000**          | 0.000**          | 0.000**          | 0.000**          | 0.000**          | 0.000**          | 0.000**          |

Unpaired 't' test

0.000 to 0.010 – significant at 1 %(\*\*)

0.01 to 0.050 – significant at 5 % (\*)

> 0.05 – not significant at 5 %

Group A= Control

Group B= Fluoride dentifrice application

Group C= Fluoride dentifrice + rinse application

There was a significant difference in the amount of fluoride released between nano-ionomeric glass ionomer and pre-reacted glass ionomer composite. There was a higher fluoride release from nano-ionomeric glass ionomer as compared with pre-reacted glass ionomer composite, irrespective of the fluoride treatment protocol which was followed throughout the study period.

**TABLE 6 DAILY FLUORIDE RECHARGE (MEAN  $\pm$  S.D) (PPM) OF PRE-REACTED GLASS IONOMER COMPOSITE AND NANO-IONOMERIC GLASS IONOMER**

| <b>Days</b>   |                  |                  |                  |                  |                  |                  |                  |                  |                  |
|---|------------------|------------------|------------------|------------------|------------------|------------------|------------------|------------------|------------------|
| <b>Materials</b>  | <b>Day 1</b>     | <b>Day 2</b>     | <b>Day 3</b>     | <b>Day 4</b>     | <b>Day 5</b>     | <b>Day 6</b>     | <b>Day 7</b>     | <b>Day 14</b>    | <b>Day 21</b>    |
| <b>PRG –Composite (Group C-Group A)</b>                 | 0.67 $\pm$ 0.35  | 0.48 $\pm$ 0.14  | 0.53 $\pm$ 0.10  | 0.624 $\pm$ 0.19 | 0.654 $\pm$ 0.15 | 0.484 $\pm$ 0.08 | 0.42 $\pm$ 0.09  | 0.538 $\pm$ 0.15 | 0.32 $\pm$ 0.11  |
| <b>Nano-ionomeric glass ionomer (Group C – Group A)</b> | 0.883 $\pm$ 0.21 | 0.943 $\pm$ 0.14 | 0.586 $\pm$ 0.16 | 0.494 $\pm$ 0.23 | 0.382 $\pm$ 0.19 | 0.406 $\pm$ 0.08 | 0.57 $\pm$ 0.15  | 0.572 $\pm$ 0.14 | 0.535 $\pm$ 0.11 |
| <b>P value</b>  | 0.089            | 0.000**          | 0.356            | 0.144            | 0.001**          | 0.036*           | 0.008**          | 0.556            | 0.000**          |
| <b>PRG –Composite (Group B-Group A)</b>                 | 0.359 $\pm$ 0.29 | 0.152 $\pm$ 0.14 | 0.087 $\pm$ 0.12 | 0.146 $\pm$ 0.17 | 0.205 $\pm$ 0.18 | 0.107 $\pm$ 0.09 | 0.207 $\pm$ 0.06 | 0.279 $\pm$ 0.08 | 0.162 $\pm$ 0.09 |
| <b>Nano-ionomeric glass ionomer (Group B-Group A)</b>   | 0.607 $\pm$ 0.16 | 0.797 $\pm$ 0.11 | 0.341 $\pm$ 0.25 | 0.294 $\pm$ 0.15 | 0.221 $\pm$ 0.06 | 0.098 $\pm$ 0.04 | 0.326 $\pm$ 0.10 | 0.348 $\pm$ 0.15 | 0.338 $\pm$ 0.07 |
| <b>P value</b>  | 0.017*           | 0.000**          | 0.004**          | 0.036*           | 0.771            | 0.757            | 0.002**          | 0.172            | 0.000**          |

Unpaired 't' test      95% confidence interval

P value

0.000 to 0.010 – significant at 1 %(\*\*)

0.01 to 0.050 – significant at 5 % (\*)

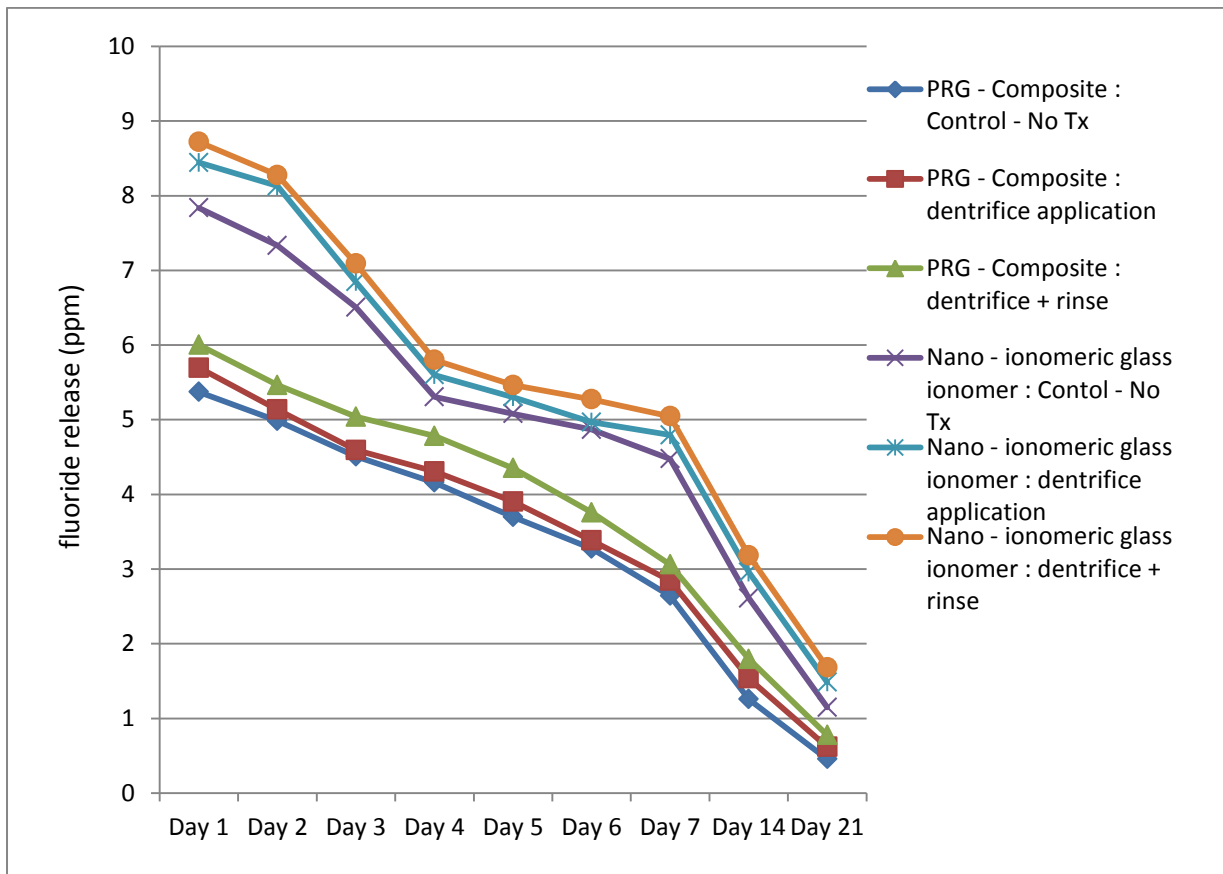
> 0.05 – not significant at 5 %



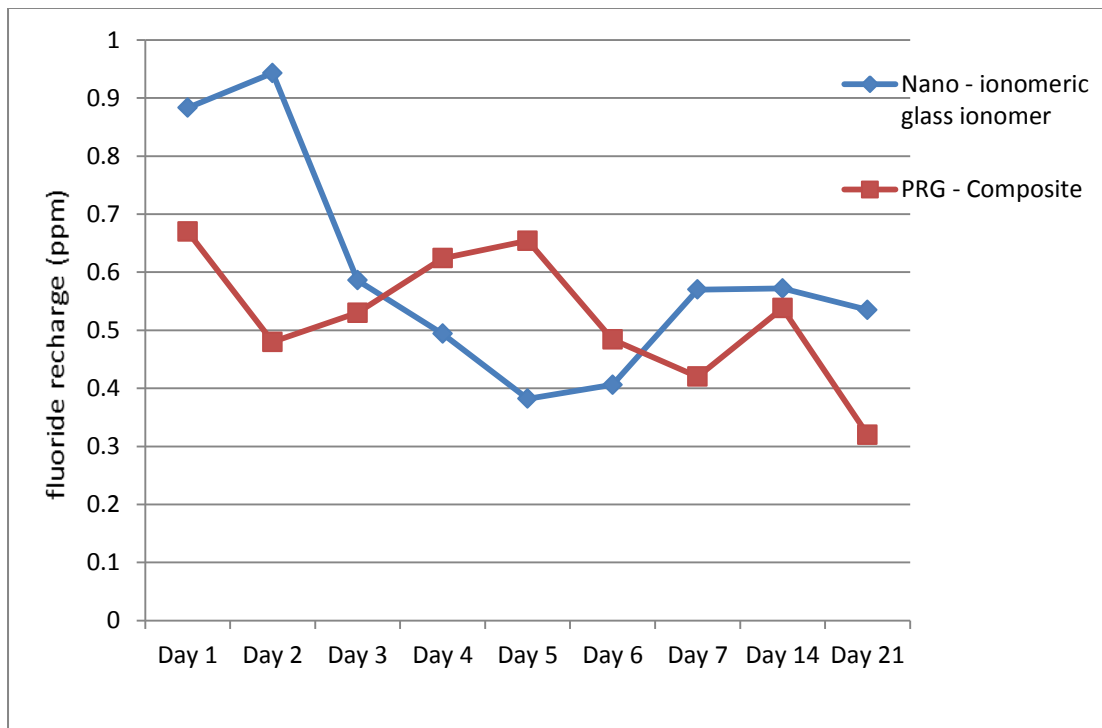
No sustained pattern of recharge was shown in both the groups. For fluoride recharge with respect to double fluoride application, the pre-reacted glass ionomer composite showed maximum recharge on day 1 and the least on day 21. In the nano-ionomeric glass ionomer group, maximum recharge was seen on day 2 and least on day 5. When compared between both the materials, nano-ionomeric glass ionomer showed greater rechargability than pre-reacted glass ionomer composite except for day 4 to day 6.

For fluoride recharge with respect to single fluoride application, the pre-reacted glass ionomer composite showed maximum recharge on day 1 and the least on day 3. In nano-ionomeric glass ionomer group, maximum recharge was seen on day 2 and least on day 6. When compared between both the materials, nano-ionomeric glass ionomer showed greater rechargability than pre-reacted ionomer composite except for day 6.

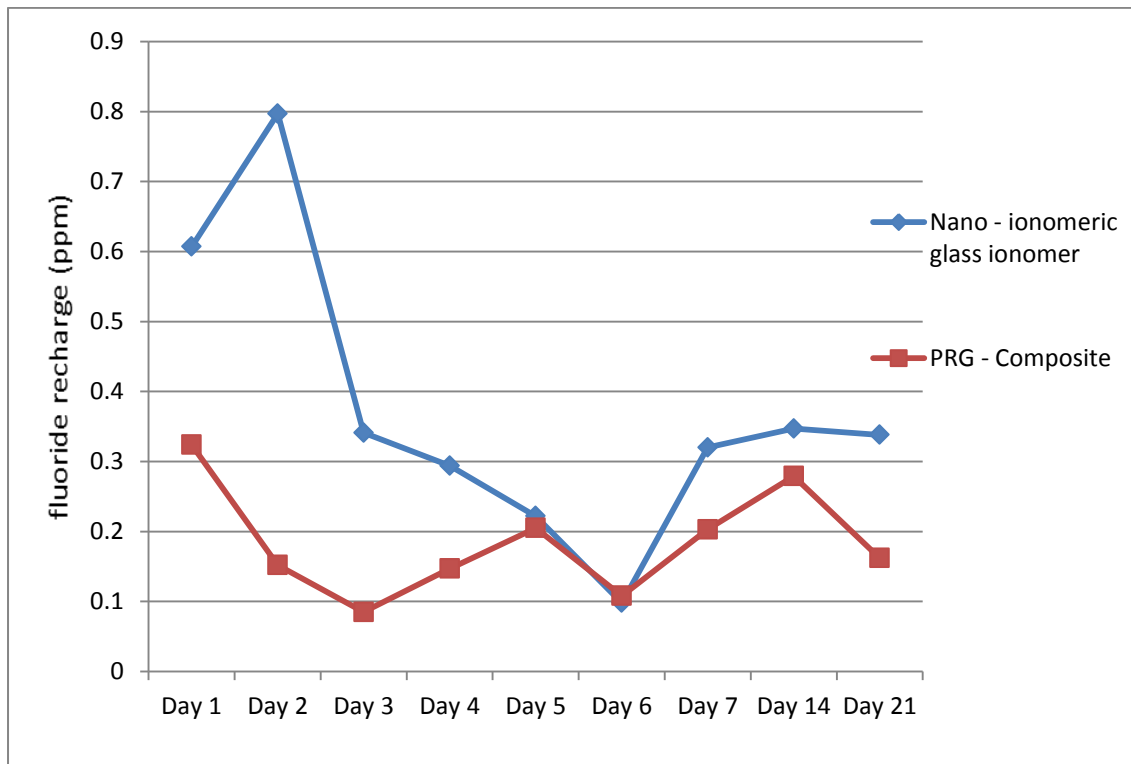
**GRAPH 1: FLUORIDE RELEASE FROM PRE-REACTED GLASS  
IONOMER COMPOSITE AND NANO-IONOMERIC GLASS  
IONOMER**



**GRAPH 2: DAILY FLUORIDE RECHARGE OF PRE-REACTED GLASS IONOMER COMPOSITE AND NANO-IONOMERIC GLASS IONOMER (GROUP C V/S GROUP A)**



**GRAPH 3:DAILY FLUORIDE RECHARGE OF PRE-REACTED  
GLASS IONOMER COMPOSITE AND NANO-IONOMERIC GLASS  
IONOMER (GROUP B V/S GROUP A)**



## **DISCUSSION**

Fluoride is a well-documented anticariogenic agent<sup>2</sup> and the release of fluoride from dental restorative materials is assumed to inhibit caries formation, progression and secondary caries initiation through a variety of mechanisms. Several restorative materials like glass ionomer cements, resin modified glass ionomer cements, compomers and giomers have received attention due to their adhesive nature, improved physical and chemical properties and their ability to release and recharge fluoride.

A recent addition to the continuum of hybrid restorative materials is the prereacted glass ionomer composite (PRG-Composite/giomer). Pre-reacted glass ionomer composites employ the use of pre-reacted glass ionomer (PRG) technology to form a stable phase of glass ionomer cement.<sup>12</sup> Manufacturers claim that the beneficial effects of glass ionomer cements are retained along with the superior physical and esthetic properties of resin composite materials.<sup>35</sup>

The development of resin modified glass ionomer restorative cements has provided tooth colored materials that have all the benefits of the glass poly-alkonate system in addition to the advantages of resin based composite materials. In 2007, a new generation of resin modified glass ionomer cement was introduced (Ketac Nano) described by the manufacturers as nanoionomer with improved physical properties. The indications for the use of nano-ionomeric glass ionomer are Class I, II, III, and V restorations, interim

therapeutic restorations for primary teeth and small Class I, III, and V restorations, transitional restorations, “sandwich” (stratification beneath bonded RBC) technique in permanent teeth.<sup>17</sup>

It has been said that the anticariogenic activity of restorative materials is linked to the release of fluoride ions which reduces the acidogenicity of plaque, thereby not favoring the growth of streptococcus mutans.<sup>5</sup> This fluoride release is dependent on various factors. Since the fluoride levels leached from fluoride containing materials decrease over time, recharging of the restoratives with fluoride has been suggested to maintain a continuously increased level of fluoride release. The ability of a restorative to act as a fluoride reservoir is mainly dependent on the type and permeability of filling material, on the frequency of fluoride exposure and on the kind and concentration of the fluoridating agent.<sup>5,36</sup> Bearing these facts in mind, the present study was undertaken to evaluate the fluoride release and recharge of pre-reacted glass ionomer composite and nano-ionomeric glass ionomer.

All the restorative specimens to be evaluated were mixed following the manufacturer’s instructions and fabricated using a standardized procedure. They were then stored in deionized water for 3 days to complete the setting reaction. The protocol for topical fluoride application was in accordance to Freedman et al which was based on the rationale that most people exposed their teeth to topical fluoride by brushing with a fluoride containing toothpaste or use a fluoride containing mouth rinse.<sup>37</sup> Fluoride release of glass-ionomers

might also be increased when application of the fluoridated dentifrice is performed by brushing of the samples instead of storage of the samples in dentifrice slurries.<sup>38</sup> The specimens were exposed to pH cycling system which was proposed Carvalho and Cury<sup>24</sup> and a fluoride ion selective electrode was used for finding the fluoride release. Fluoride ion selective electrode is well documented and an accepted procedure.<sup>37,39-41</sup>

The results of the present study show that fluoride release was influenced by immersion media for both the materials regardless of the fluoride treatment regime (tables 1, 2 & 3). Fluoride release was greater in demineralization solution when compared to the release in the remineralization solution ( $P=0.000^{**}$ ) for both the pre-reacted glass ionomer composite and nano-ionomeric glass ionomer irrespective of treatment protocol throughout the study period, even though the specimens were immersed in the demineralizing solution for only one third as long as in the remineralizing solution. The increasing amount of fluoride in acidic media could be explained by the fact that a decrease in pH increases the dissolution of the material leading to a higher fluoride level in the acidic immersion.<sup>15,41-44</sup> Another explanation for the higher fluoride release in acidic media is that fluoride is principally used as a flux in the manufacturing process and is incorporated into the glass component. Upon mixing the glass powder with polyalkenoic acid, the fluoride ions are released by the initial attack on the

surface of the glass particles. Fluoride is not a matrix-forming species and takes no further part in the setting reaction, but remains within the matrix.<sup>35</sup>

In the present study (table 4), although there was a significant difference in the amount of fluoride released during the entire study period, there was an initial high release during the first two days which was then followed by a rapid reduction in the rate of release of fluoride till day 7 followed by a gradual reduction till day 21 for both the materials in either of the solutions irrespective of treatment protocols. This initial rapid release of fluoride on the first two days is termed as “fluoride burst.”<sup>2,14,45</sup> This is in contrast to the findings of Yap et al.,<sup>14</sup> who found no initial fluoride burst effect with pre-reacted glass ionomeric composite. However, fluoride release from aged and refluoridated specimens mostly did not reach the initial fluoride release of the material.<sup>14,46</sup>

In pre-reacted glass ionomer composite and nano-ionomeric glass ionomer, there was a significant difference in the amount of fluoride released with respect to increase in the fluoride supplementation compared to control group throughout the experimental period. With respect to pre-reacted glass ionomer composite, similar findings were reported by Dhull and Nandalal<sup>3</sup> and by Itota et al.<sup>13</sup> We could not come across studies where the effect of different fluoride treatment regimens on nano-ionomeric glass ionomer was evaluated. As cited in the review by Weigand et al.,<sup>2</sup> fluoride release after application of fluoridated agents may occur partly by washout of fluoride ions that are



retained on the surface or in the pores of the restorative. Surface bound fluoride might be more easily detached during an acidic attack, such as in erosion. Also, free fluoride incorporated into the matrix might be washed out.<sup>47</sup> Glass-ionomers are mostly found to have significantly better capability to act as a fluoride reservoir than composite resin-based materials. This fact can be explained by the loosely bound water and the presence of solutes in the porosities in the glass-ionomer, which may be exchanged with an external medium by passive diffusion.<sup>48,49</sup> The absorption and re-release of fluoride might be determined by the permeability of the material. Thus, a completely permeable substance could absorb the ions deep into its bulk; while a relatively impermeable material can only absorb fluoride into the immediate subsurface.<sup>5</sup>

(Table 5) There was also a significant difference in the amount of mean fluoride release between nano-ionomeric glass ionomer and pre-reacted glass ionomer composite with higher fluoride release from nano-ionomeric glass ionomer ( $P=0.000^{**}$ ) compared to the pre-reacted glass ionomer composite irrespective of the treatment protocol throughout the study period. The difference can be possibly explained as the nano-ionomeric glass ionomer contains nano-sized filler particles, which provide a larger surface area, thereby increasing the acid-base reactivity, and hence, has the capacity to release fluoride from the powder more quickly, increasing the fluoride release of the material.<sup>31</sup>

In the present study (Table 6), no sustained pattern of recharge was noticed among both the materials. In the treatment group where fluoride dentrifice and rinse were used, the pre-reacted glass ionomer composite showed maximum recharge on day 1 and least on day 21 whereas nano-ionomeric glass ionomer showed maximum rechargability on day 2 and least on day 5. Nano-ionomeric glass ionomer showed significantly higher amount of Fluoride recharge than pre-reacted glass ionomer composite except for days 4-6.

For the treatment group involving fluoride mouthrinse, the pre-reacted glass ionomer composite group showed maximum recharge on day 1 and the least on day 3, whereas for the nano-ionomeric glass ionomer group, maximum recharge was seen on day 2 and the least on day 6. Nano-ionomeric glass ionomer showed significantly higher amount of Fluoride recharge than pre-reacted glass ionomer composite except for day 6.

In general, materials with higher initial fluoride release have higher recharge capability.<sup>46,47,48</sup> The Daily exposure of filling materials to fluoridated dentifrices has demonstrated a high rechargability for glass ionomers, while resin based materials have demonstrated a negligibly small amounts of replenishment.<sup>36,41,50</sup>

The present study is an attempt to find out the fluoride release and recharge from two recent restorative materials claimed by the manufacturers to have superior physical and chemical properties with higher fluoride releasing

capacity and wide range of application in pediatric restorative dentistry. A major limitation of our study was that, since this being an in-vitro study, the fluoride release profiles of the materials may not be comparable with in-vivo conditions, as the higher ionic concentration of saliva, or presence of pellicle would influence the ion release rate from these cements.<sup>44</sup> It is also important to point out that the extrapolation of the laboratory findings to the in-vivo performance of the restorative materials should be done carefully, as the conditions between both situations are different.<sup>32</sup>

Within the limitations of the present study, it can be concluded that nano-ionomeric glass ionomer showed better fluoride release and recharge with higher amount of fluoride release into demineralizing solution than remineralizing solution. Also fluoride supplementation increases the uptake and release of fluoride ions of the study materials. The fluoride releasing capacity decreases over a period of time. Future studies should be aimed towards controlled clinical trials with more complex experimental designs comprising of large number of factors which influence the properties of dental materials in real clinical situations to draw valid conclusions.

## **CONCLUSION**

1. The mean fluoride release of pre-reacted glass ionomer composite was significantly greater when the test specimens were immersed in the demineralizing solution than in the remineralizing solution, even though the specimens were suspended in the remineralizing solution for thrice the duration than compared to specimens suspended in the demineralizing solution.
2. The mean fluoride release of nano-ionomeric glass ionomer was significantly greater when the test specimens were immersed in the demineralizing solution than in the remineralizing solution, even though the specimens were suspended in the remineralizing solution for thrice the duration than compared to specimens suspended in the demineralizing solution.
3. The two restorative materials demonstrated decreased amount of fluoride release over the course of the study.
4. Fluoride release was related to the dose of daily supplemental fluoride which was applied.
5. Fluoride release was greater from samples exposed to both fluoride dentrifice and mouth rinse. Similarly, fluoride release was also found to be significantly greater in the fluoridated dentrifice group when compared to the control group.

6. Nano-ionomeric glass ionomer showed better amount of fluoride release than pre-reacted glass ionomer composite throughout the course of the experiment period irrespective of the fluoride treatment supplementation.
7. No sustained pattern of fluoride recharge was shown by both the restorative materials, even though the nano – ionomeric glass ionomer showed better rechargability then pre – reacted glass ionomer composite.

## **SUMMARY**

This in-vitro study was designed to investigate the effects of daily fluoride exposures on fluoride release and recharge by pre-reacted glass ionomer composite (Beautifil II<sup>TM</sup>, Shofu Inc.) and nano-ionomeric glass ionomer (Ketac<sup>TM</sup> N100, 3M ESPE).

Seventy two specimens (36 of each material) were prepared and cured as per the manufacturers' recommendations by placing the restorative materials into a Teflon mold (5 mm x 2mm). Each specimen was subjected to one of three daily treatments (n=12): (1) no fluoride treatment (control); (2) application of a fluoride dentifrice (1000 ppm) for one minute once daily; (3) the same regimen as (2), plus immersion in a 0.05% sodium fluoride (NaF) mouth rinse (225 ppm) for one minute immediately following the dentifrice application.

Each specimen was suspended in a storage vial containing 10 ml demineralizing solution (pH 4.4) at 37°C for six hours, then transferred to a new test tube containing 10 ml remineralizing solution (pH 7.0) at 37°C for 18 hours. Fluoride treatments of the specimens were completed every day, prior to their immersion in the demineralizing solution. Media solutions were buffered with equal volumes of TISAB II; fluoride levels were measured using a digital ion analyzer and fluoride electrode. Fluoride release was measured throughout the 21 day duration of the experiment.

Fluoride release of pre-reacted glass ionomer composite and nano-ionomeric glass ionomer was influenced by immersion media. Regardless of the fluoride treatment, the mean fluoride release was significantly greater when test specimens were immersed in the demineralizing solution than in the remineralizing solution. The nano-ionomeric glass ionomer showed a better amount of fluoride release than pre-reacted glass ionomer composite during the course of the experiment period, irrespective of the fluoride treatment supplementation. The additional fluoride supplementation improved the fluoride release and recharge ability for both the materials when compared to their respective control groups. An initial rapid release of fluoride was noticed for both the pre-reacted glass ionomer composite and nano-ionomeric glass ionomer during the first two days. This initial rapid release is termed as “fluoride burst”. The fluoride recharge for both materials did not show any sustained pattern of release. When compared between both materials, nano-ionomeric glass ionomer demonstrated a greater ability to recharge than compared with that of pre-reacted glass ionomer composite.

However, controlled clinical trials with experimental designs comprising of a large number of factors which influence the properties of dental materials in real clinical situations are needed in order to draw valid conclusions.

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